

**The Journal**  
*of the*  
**SOCIETY OF DYERS**  
**AND COLOURISTS**

Volume 68 Number 12

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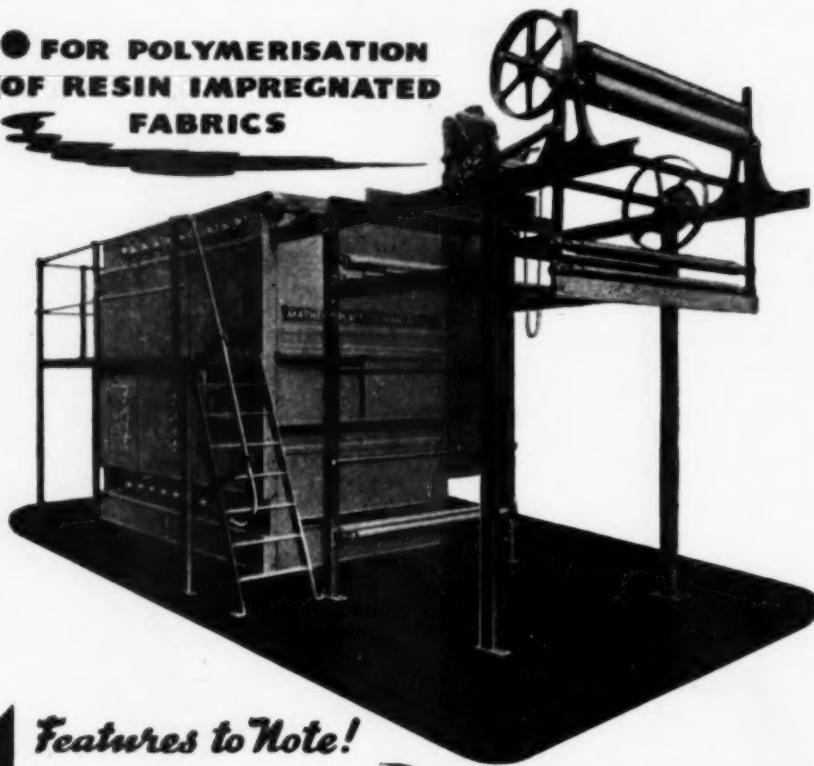


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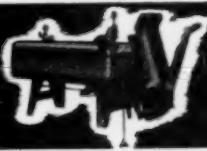
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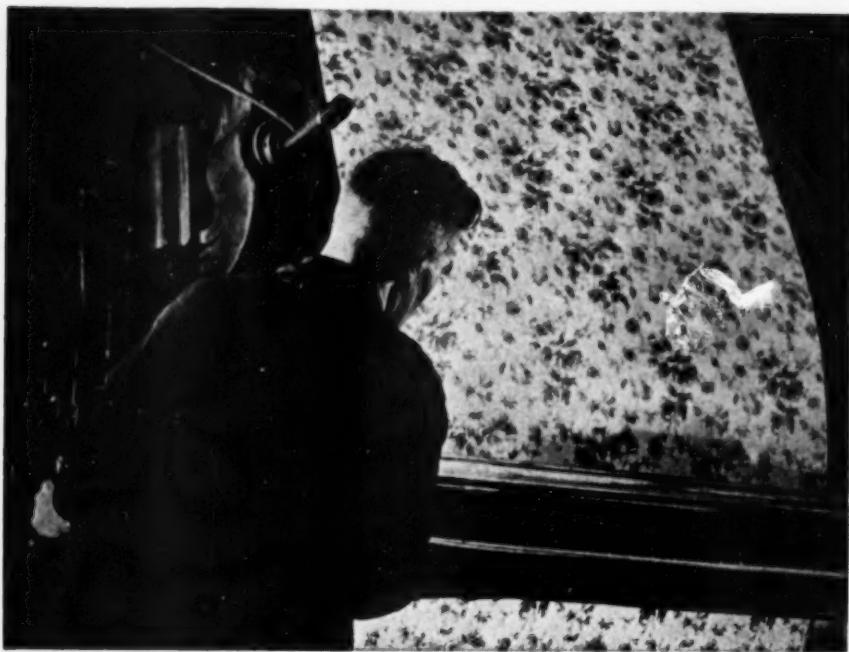
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## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1952 and pages 237-240 of the July 1952 issues of the *Journal*, or write to *The General Secretary, The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire (Telephone Bradford 25138-9)*. *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURE

The Place of the Technologist in our Industry

*F. E. Goodall*

### COMMUNICATIONS

Zacharie Roussin

*R. Brightman*

The Influence of Particle Size on the Dyeing Properties of Vat Dyes

*T. Flanagan*

New Methods of Analysis for Dyes and Intermediates

*C. H. Giles et al.*

### MEMBERS' CHANGES OF ADDRESS

Labnowski, J. M., formerly of 86 Musters Road, West Bridgford, to 62 Thornewood Rise, Nottingham

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## Forthcoming Meetings of the Society (see also page xxv)

### MANCHESTER SECTION

Unless otherwise stated meetings are held in the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

1953	
Friday 16th Jan.	G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). <i>The Measurement of Colour</i>
Friday 20th Feb.	W. Speke, Esq., B.Sc. (I.C.I. Ltd., Dye-stuffs Div.). <i>High Temperature Dyeing of Viscose Rayon</i>
Friday 13th March	Manchester College of Technology. Afternoon and Evening Symposium on <i>New Fibres</i> . Details to be announced later
Friday 20th March	Joint meeting with the British Association of Managers of Textile Works. Midland Hotel, Manchester. Lecture by Dr. Fargher (Shirley Institute). Details to be announced later
Friday 17th April	ANNUAL GENERAL MEETING. Details of lecture to be announced later

### NORTHERN IRELAND SECTION

All Meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.

1953	
Wednesday 14th Jan.	E. R. Wiltshire, Esq. (I.C.I. Ltd.). <i>The Control of Azoic Dyeing Processes</i> (Joint meeting with Foremen Dyers Guild)
Wednesday 11th Feb.	F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.). <i>Pressure Dyeing and Bleaching with Possible Indication of New Developments</i>
Wednesday 11th March	Dr. B. C. Gee. <i>The Educational Side of Textile Processing</i>
March/April	ANNUAL GENERAL MEETING and DINNER (date will be announced later)

### LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1953	
Friday 9th Jan.	Uses of Microscopy in Textile Dyeing and Finishing (Clayton Aniline Co. Ltd.).
Friday 6th Feb.	F. Ward, Esq. (Courtaulds Ltd.). <i>Methods and Principles of Textile Fireproofing</i>
Friday 6th March	Title to be announced later

### MIDLANDS SECTION

Except when otherwise stated, Lecture Meetings commence at 7 p.m.

1953	
Wednesday 28th Jan.	J. Boulton, Esq., M.Sc.(Tech.), F.R.I.C., F.T.I. <i>Modern Dyeing Theory for the Plain Man</i> . Masonic Hall, Hinckley, 7.30 p.m. (Joint meeting with the Textile Institute and Hinckley Textile Society)
Thursday 19th Feb.	E. M. Walker, Esq., F.T.I., F.R.M.S. <i>The Chemist and Technologist in the Hosiery and Knitted Goods Industry</i> . (Joint meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster, 7.30 p.m.

### Midlands Section—continued

Wednesday 29th Feb.	E. R. Wiltshire, Esq., B.Sc. <i>Azoic and other Fast Dyes on Knitted Cotton Fabric</i> . College of Technology, Leicester
Friday 13th March	MIDLANDS SECTION DINNER. Royal Hotel, Leicester
Wednesday 18th March	G. H. Osborn, Esq., F.R.I.C., A.M.Inst. M.M. <i>Colour in Chemistry</i> (Joint meeting with the British Association of Chemists). School of Arts and Crafts, Derby
Wednesday 22nd April	ANNUAL GENERAL MEETING OF THE SECTION. Gas Board Theatre, Nottingham

Towards the end of the session Dr. H. White (Textile Research Institute, Princeton, U.S.A.) will lecture in Nottingham. Particulars later

### HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Huddersfield, at 7.30 p.m., unless otherwise stated

1953	
Tuesday 20th Jan.	G. H. Rostron, Esq., A.R.I.C. (J. Crosfield). <i>Textile Process Soaps</i>
Tuesday 17th Feb.	INTER-SECTIONAL DISCUSSION
Tuesday 17th March	Dr. H. A. Thomas (Courtaulds). <i>The Rôle of Dyeing, Printing, and Finishing in Fabric Development</i>
Tuesday 7th April	ANNUAL GENERAL MEETING followed by a lecture—Dr. J. F. Gaunt. <i>A Study of the Aftercrown Process of Wool Dyeing</i>

### SCOTTISH SECTION

All meetings at St. Enoch Hotel, Glasgow, 7 p.m., unless otherwise stated

1953	
Tuesday 13th Jan.	J. C. Brown Esq. (Clayton Aniline Co. Ltd.). <i>Microscopy for the Dyer and Finisher</i>
Tuesday 17th Feb.	J. Barr Esq. (Turnbull's Ltd.). Details later
Tuesday 17th March	ANNUAL GENERAL MEETING, to be held at the ST. ENOCH HOTEL, GLASGOW at 7 p.m. To be followed by <i>Brains Trust</i> at 7.30 p.m. The Panel is to be formed from Sectional Members and details will be circulated later. By courtesy of the College Authorities an invitation has been extended to inspect the new laboratories on this occasion

### MANCHESTER JUNIOR BRANCH

All meetings commence at 6.30 p.m. in the Reynolds' Hall, College of Technology, Manchester.

1953	
Monday 2nd Feb.	Dyeing and Finishing. <i>The Public's Reaction to Fastness Properties</i> J. S. Ingham Esq.
Monday 2nd March	Title to be given later.

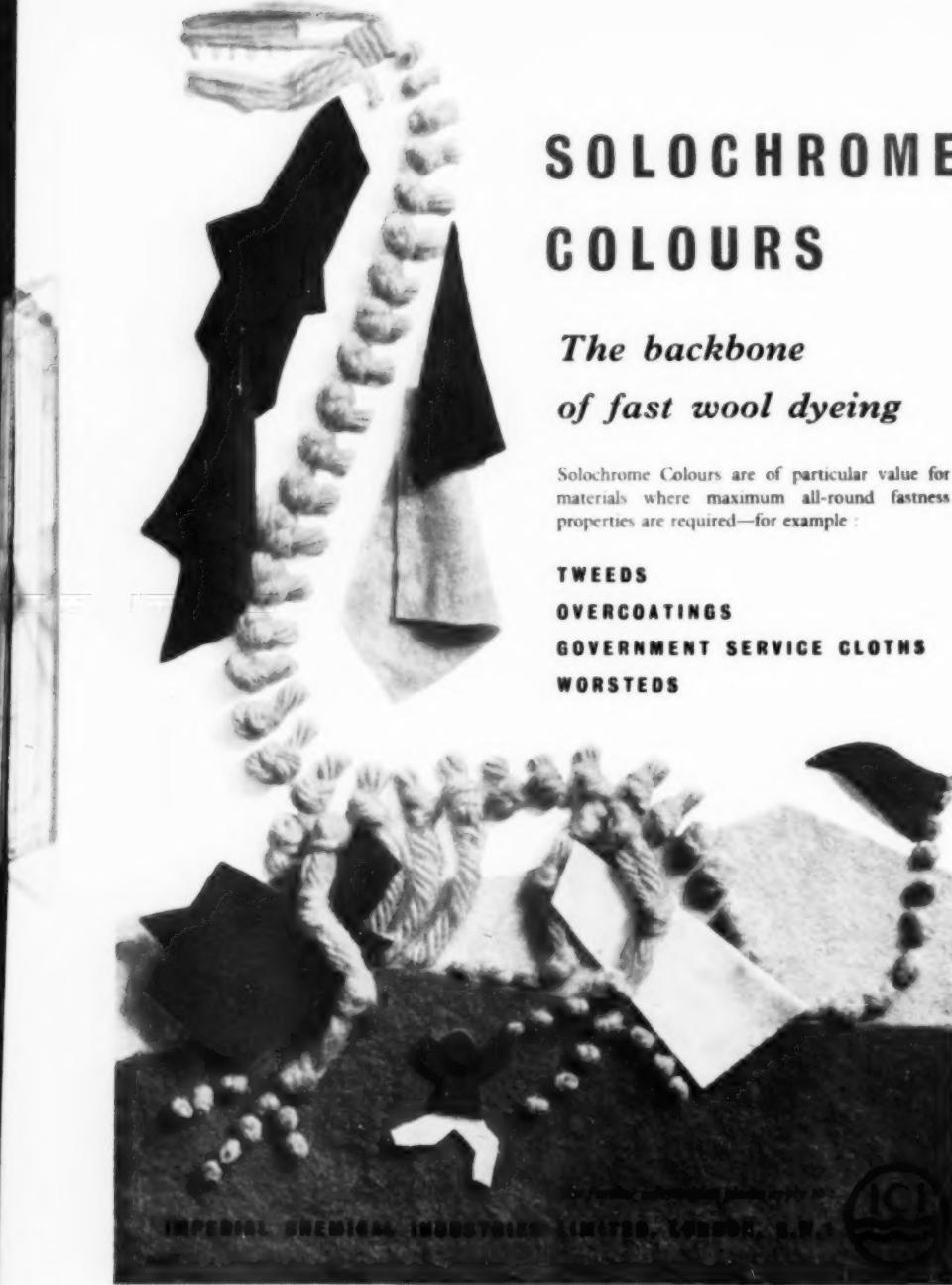
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# THE JOURNAL OF THE Society of Dyers and Colourists

Volume 68—Number 12

DECEMBER 1952

*Issued Monthly*

## Proceedings of the Society

### The Ninth John Mercer Lecture Non-felting Wool and Wool Mixtures

FREDERICK CHARLES WOOD

*Meeting held at the College of Technology, Leicester, on 9th May 1952, Mr. H. H. Bowen,  
President of the Society, in the chair*

Hagiography has a large and interesting literature, and the achievements of the saints, martyrs, prophets, and seers are rightly celebrated in ethical societies who value their historicity.

The reverence which is given to those who have excelled in ethical science (theoretical and practical) is certainly not a misplaced admiration, for we all know that ethics is naked before material power, which throughout the ages seems to get more mixed with pragmatism and materialism and expediency. Their achievements against fearful odds are, therefore, all the more astounding.

Science and technology have their hagiography, and our Society of Dyers and Colourists has rightly recommended and commemorated the name of John Mercer for veneration. He was, as the Greek has it, *έθεος* — inspired, god-filled — or in English "enthusiastic", from the verbal form *έθεω*—*ω*.

Mercer himself said that, on seeing the beautiful orange colour of the dress of his little step-brother sitting on his mother's knee, he became all on fire to learn dyeing<sup>1</sup>.

Hagiographic studies of scientists should not merely be concerned with drab historical events in the advances made in our technologies, but also with the urges or fires, lit whence we dimly know, which lead to discovery and invention. These urges, fires, and shafts of inspiration are at least as obscure as are the processes discussed in religious hagiography.

One difference between religious and scientific hagiography is apparent to the student who finds that in the latter there is no "devil's advocate" to ascertain the purity of the inventor's intention, and that he never has to go through the stages of veneration, blessedness, sainthood. A scientist who becomes the subject of a memorial lecture may have played the very devil with his contemporary

colleagues or even with his wife, but all this is passed over, and we praise only his scientific acumen and achievement. John Mercer was in this respect not two-faced but by report a very genial fellow, and his good works other than in science are well worth reading about and emulating. Could not Parnell's *Life*<sup>2</sup> of the father of textile chemistry be republished, so that our younger members may gain some inspiration, and indeed we older members may reflect on the possibilities of useful inventions for us still to make?

I mention and include the older members because intelligence and superior achievement are not a simple function of age. Psychological writers may and do quote curves to show that the peak for great scientific, inventive, or musical achievement falls in the thirties<sup>3</sup>. Gentlemen, do not believe it—John Mercer was 59 when he took out his patent for "mercerisation" in 1850. He was right off this curve, and I for one refuse to be fitted on the curve, and I hope you do, too. Do not let statistics hypnotise you into thinking that you are done for at 40, 50, or 60 years of age.

I suppose new discoveries and inventions arise by the integration of the fractional knowledge we all possess in the mind's recollection of its own experiences coupled with the experiences of others written down in our journals, but I am not at all persuaded that this is the complete mechanism by which we work towards new ideas and inventions.

As I have said elsewhere<sup>4</sup>, if we would take the trouble to put down for ourselves and for posterity "the truth, the whole truth, and nothing but the truth", we might speed up the production of discoveries and inventions, and maybe realise the importance of the apparently trivial.

There are both positive and negative catalysts in this invention business, and I make no apology for bringing this up in this introduction, because

every student after graduation may expect to come right up against this problem of speeding up his work if he aspires, either singly or as a member of a team, to make his mark in the textile research field. He must vigorously eliminate the negative "catalysts", whether of animal (human), vegetable, or mineral origin. We must face the truth that our minds are imperfect instruments, and cannot scan the whole field of a subject as the scanning ray in television. We find it difficult enough to formulate, let alone grasp, an integrated picture which shall be completely balanced and in focus. This can be corrected to some extent by wisely directed team search as well as team research, for, as Goethe wrote, "Nothing is so fruitful as ignorance in action".

In the subject I am to discuss there are a great many facts known only to some of us. Other facts are distorted, and we are apt to put a bias on the wood, so that it will go the nearest to the jack—or should it be jackpot! The chief item which is subject to wishful thinking in non-felting wool is the degree of alteration of the handle of the material brought about by the processes.

I take it that the Society has invited me to deliver this memorial lecture because my colleague, A. J. Hall, and I were the first to demonstrate a highly successful method of treating wool with caustic soda ninety years after Mercer had applied it to cotton. This work of Hall and myself was carried out in October 1939 in the laboratories of the Tootal Broadhurst Lee Co. (T.B.L.), and the process is successfully operating on a large scale<sup>4</sup>. After only three days' work we were able to draft a patent application.

An adequate discussion of the subject of non-felting wool and wool mixtures would require a special monograph in order to do justice to the workers and to the vast amount of painstaking and elegant research which has been carried out during the last decade. All I can attempt to do in the hour at my disposal is to survey some aspects of the subject, since during the last three years many summaries have appeared in which both theoretical and practical questions have been discussed. Not least is the *Review of Textile Progress*, published by the Society and the Textile Institute jointly, worthy of special notice<sup>5</sup>.

The problem of reducing or eliminating the shrinkage of wool by felting is one aspect of the great general problem of textile clothing, viz. that of stability of shape and size in wear and wash.

The crease-resist process of Foulds, Marsh, and Wood<sup>6</sup> dealt at bottom with another aspect of the same problem: it procured stability to crushing and creasing of cellulose materials, and "inaugurated a new era in the finishing of textile materials" according to Lipson and Speakman<sup>7</sup>.

Up to about 1936 only the elementary facts of felt-resist processes were known, and these were mainly concerned with treatment by the halogens chlorine and bromine.

This stagnant period seemingly ended with the publication of the Hall, Hickling, and Pentecost patent on the use of sulphuryl chloride in white spirit<sup>8</sup>.

Thereafter, a great deal of attention was given to the subject, both scientifically and technically, so that to-day there are, according to Teplitz<sup>9</sup>, over sixty processes "on the books", though it is not known how many of these laboratory methods have succeeded on the large scale.

Speakman, Stott, and Chang stated<sup>10</sup> that, for milling shrinkage to take place, the fibre must—

- (1) Possess a surface structure
- (2) Be capable of stretching and deformation
- (3) Possess the power of recovery from stretching.

The cure of felting has been effected by methods which may very roughly be summarised as follows—

- (1) Partial or complete removal of surface scales by oxidation, halogenation, enzyme action, etc.
- (2) Modification, without removal, of the scale surface, e.g. by means of alkali in non-aqueous solvents, mixed hypochlorite and permanganate
- (3) Masking of the scale structure by extraneous addition, e.g. of highly polymeric substances
- (4) Alteration of the elastic properties of the fibre by internal deposition of polymerisable substances (monomers), e.g. ethylene sulphide in presence of water vapour<sup>10a</sup>
- (5) Bonding of fibres in contact (spot welding effect), e.g. bisalkoxymethylureas (bis-a-butoxymethylurea)<sup>11</sup>; melamine and anhydrocarboxylglycine are further examples.

Processes involving the use of halogens and halogen compounds are seen to be very numerous from the following list—

- (1) Solid bleaching powder diluted by an inactive finely divided substance such as soda ash or silica, etc.<sup>12</sup>.
- (2) Gaseous chlorine on wool at normal moisture contents in which chlorine is continuously fed into a chamber at normal pressure, and water removed<sup>13</sup>.
- (3) Gaseous chlorine at lower controlled moisture content in an evacuated chamber<sup>14</sup>.
- (4) Chlorine dissolved in organic solvents, e.g. carbon tetrachloride.
- (5) Organic hypochlorites, e.g. methyl and butyl hypochlorites, as vapours or dissolved in organic solvents.
- (6) Vapours of inorganic acid chlorides such as nitrosoyl, sulphuryl, and chromyl chlorides.
- (7) Aqueous hypochlorites of alkali metals or alkaline-earth metals—
  - (a) At pH 8.5-9.0<sup>15</sup>.
  - (b) Short-time (2-10 sec.) treatment at pH 7.5-9.0 with solutions containing 1-6% active chlorine<sup>16</sup>.
  - (c) At controlled pH and rH in presence of formic acid at lower temperatures, etc.<sup>17</sup>.

- (d) Chlorination controlled by means of chloro-nitrogen compounds, e.g. chlorosulphamic acid in presence of common salt<sup>18</sup>.
- (e) Hypochlorite mixed with permanganate<sup>19</sup>.
- (f) 1-6-75% Available chlorine is applied to fabric for 5 sec. only under "no reaction" conditions. The temperature is then raised to 200-220° F. for not more than 15 min. Minimum damage and maximum non-shrink properties are claimed<sup>20</sup>.

Oxidising agents not containing halogens have been proposed—

(1) Treatment is carried out with a very dilute solution of potassium permanganate under 5-10 lb. per sq. in. pressure above 100° C. It is claimed that the wool is bleached and rendered non-felting and level dyeing<sup>21</sup>.

(2) Peracids in carbon tetrachloride<sup>22</sup>.

(3) The Lanfix process — 2-vol. hydrogen peroxide plus copper catalyst at pH 10.5 and 50° C. for 1 hr.<sup>23</sup>.

Caustic alkalis have been successfully applied, the following being the principal methods—

(1) Substances having an alkaline reaction in solvents such as aliphatic alcohols—methyl, ethyl, propyl, butyl<sup>24</sup>.

(2) Alkaline substances in solvents for the alkali diluted with non-polar non-solvents for the alkali, e.g. 0.6% sodium hydroxide in 10% (by vol.) n-butanol plus 90% white spirit<sup>25</sup>.

(3) Ethyl alcoholic potash and water, etc.<sup>26</sup>.

The greatest activity of all has been directed to the application of highly polymeric substances either as monomers or as partly condensed polymers. Only a selection of the many proposals can be given here—

(1) Urea- or melamine-formaldehyde, particularly when alkylated<sup>27</sup>.

(2) Methylated hydroxymethylmelamine used to impregnate wool before spinning and weaving, after which curing is effected in sulphur dioxide<sup>28</sup>.

(3) Aqueous urea- or melamine-formaldehyde condensation products are dried into wool, and then curing is effected in steam containing a volatile acid<sup>29</sup>.

(4) Bis-N-butoxymethylurea in alcoholic solution<sup>30</sup>.

(5) Diisocyanates such as *m*-phenylene diisocyanate in white spirit<sup>31</sup>.

(6) *m*- and *p*-Vinylphenyl isocyanate copolymerised with styrene<sup>32</sup>.

(7) Maleic anhydride-alkyl acrylate copolymer. Increased strength and soft handle claimed<sup>33</sup>.

(8) Silicones<sup>34</sup>. Less than 5% increase in weight is required, as with anhydrocarboxyglycine. Si-NH linkages are supposed to be formed.

(9) A copolymer of 10-100% of ethoxyethyl acrylate and 0-90% of ethyl acrylate, then cured at 200-300° F. Increased tensile strength and a soft handle are claimed<sup>34</sup>.

(10) 3-30% of a monomer of a thermosetting resin such as an alkylated hydroxymethylmelamine

is applied and heated in two stages at 100-160° F. and then for 15 min. at 200-240° F.<sup>35</sup>.

(11) Polymerised butadiene mixed with a urea- or melamine-formaldehyde polymer. The latter corrects the sticky feel imparted by the former<sup>36</sup>.

(12) An aqueous emulsion of 5% of a chloroprene polymer with a wetting agent or a copolymer with butadiene, styrene, or an alkyl acrylate<sup>37</sup>.

(13) Anhydrocarboxyglycine in ethyl acetate<sup>38</sup>.

(14) *N*-Carboxyanhydrides of amino acids, including (13) above<sup>39</sup>.

(15) Methacrylic acid and hydrogen peroxide<sup>7</sup>.

(16) Ethylene sulphide with<sup>40</sup> or without water.

(17) Methacrylamide<sup>41</sup>.

(18) Infrared or radio-frequency heating is used with an aqueous solution or dispersion of a resin-forming material and an acid catalyst<sup>42</sup>.

(19) 3:4-*iso*Propylidene-1:2-5:6-dianhydro-mannitol for 24 hr. at 50° C.<sup>43</sup>.

(20) Polymerisation products from ethylene and carbon monoxide containing a long-chain-CH<sub>2</sub>-CH<sub>2</sub>-CO- linkage system are reduced and aminated with hydrogen and ammonia or a primary or secondary amine; they have been proposed for imparting water-repellent properties to fabrics, for reducing the shrinkage in wool fabrics, and also rendering acid dyes fast to water<sup>44</sup>. The process involves condensation under 600 atm. for 15 hr. at 150° C., during which time the reactants are agitated in presence of a nickel hydrogenation catalyst. The product is dissolved in toluene and filtered to remove the catalyst, and then the toluene is removed by distillation followed by further removal of toluene at 90° C. for 2 hr. at reduced pressure. The light straw-coloured liquid, a polyamine, dissolves readily in 5% aqueous acetic acid to give colourless solutions.

For application to wool the polyamine is dissolved in 6% aqueous acetic acid by heating to 90° C. On cooling, formaldehyde is added to give a mixture containing 5% polymer, 5% acetic acid, and 3.5% formaldehyde. Wool is impregnated with this solution, squeezed to remove excess liquor, and then pressed between paper towels for uniform treatment and heated at 100° C. for 15-30 min., or after air drying for 24 hr. or longer.

A treated sample shrank 3-5% in area as compared with 25-35% shrinkage of the untreated material. The handle and other properties are reported to be unchanged<sup>45</sup>.

In this specification<sup>46</sup> a treated sample had 3% relaxation and 4% felting shrinkage, compared with the untreated values of 10% and 40% respectively.

All these processes operate and are explicable by one or more of the ideas outlined above.

All commercial processes function according to Alexander<sup>47</sup> by two mechanisms—

(1) By removing or decreasing the directional frictional effect (D.F.E.) either by raising the with-scale coefficient or by lowering the anti-scale frictional effect. These changes in the D.F.E. are

connected with the action of the reagent on the scale tips and also on the epicuticle.

(2) By gluing together the fibres within the yarns, thus preventing fibre movement.

Other writers have drawn attention to Speakman's contention<sup>45</sup> that some processes can be explained by modification of the elastic properties of the fibres.

The best and cheapest of the alkali processes disclosed by T.B.L. Hall, and Wood makes use of a preponderating amount of a (non-polar) non-solvent for alkali, generally white spirit, and the method outlined in the specifications<sup>46</sup> has the following advantages—

(1) Simplicity of preparation of the caustic soda-butanol-white spirit reagent, which contains about 0.6% of sodium hydroxide, its saturation value (the effectiveness of this reagent was discovered by the lecturer on 20th November 1939).

(2) This solution may be used over and over again, merely requiring recausticising without frequent distillation. The same solution may be used over 100 times.

(3) When distillation is required, only a very small unit is necessary.

(4) Wool of ordinary moisture content, generally 12-18%, gives the optimum results.

(5) The reaction is isothermal, for no heat is developed or absorbed in the treatment.

(6) Treatment takes place at ordinary temperatures.

(7) Each fibre receives treatment by virtue of the low viscosity and low surface tension of the solution.

(8) The effective high polymer used to mask the scale surface is the wool protein itself, which is already joined to the fibre.

(9) The scale surface at the tips is slightly masked and is not removed.

(10) Practically all mixtures of wool with other fibres may be treated as a final finishing treatment without damage, e.g. wool-rayon, wool-cotton, wool-nylon, wool-linen.

(11) Nothing is added to the wool and nothing is taken away.

(12) Softness of handle is retained by virtue of the very small amount of masking material.

(13) No high baking temperature or cure is required.

(14) The products have a clear finish which does not go fuzzy or hairy during their life.

(15) The non-felt finish is permanent, and does not break down during the wearing and washing life of the material.

Whilst the application of caustic alkali to cotton goods associated with the famous name of Mercer is so well known and is utilised in the textile industry and other fields, no technical progress was made in its application to wool, owing to its extremely rapid and destructive action when applied in obvious conventional ways, until 1939, when Hall and Wood began their work. The high viscosity of strong solutions of caustic soda makes their application to wool very difficult—firstly

because of the difficulty of penetration of the solution to each fibre, and secondly because, once application has been made, such a large amount of alkali is attached to the wool that it is practically impossible to remove it without ruining the wool.

The comparative viscosities and surface tensions of some non-aqueous solutions measured during the course of our researches are given in Table I. It will be seen that, whereas strong aqueous caustic alkali has a viscosity of 73 centipoises at 20°C., a dilute alkali solution in 10% butanol + 90% white spirit has a viscosity of 1.2 centipoises, about equal to that of water.

The very destructive action of aqueous alkali on wool is so well known that one need only refer to Jager's work in 1934<sup>47</sup>.

One or two previous workers have carried out simple experiments to find the acidity of wool by exposing stoved wool to dilute solutions of potash in ethyl alcohol for 24-336 hr. (Stepanow 1900)<sup>48</sup>.

TABLE I  
Viscosity and Surface Tension of Alkalies dissolved in Non-aqueous Media

Solution	Viscosity (centipoises)	Surface Tension 0°C., 20°C.	(dynes/cm.)
Water ... ... ...	—	1.00	73
50% Aqueous NaOH ...	—	73.0	—
White spirit ... ...	1.52	1.08	29.6
Paraffin ... ...	2.46	1.64	30.9
n-Butanol ... ...	5.3	2.9	27.5
10% Butanol + 90% white spirit ...	1.6	1.10	30.4
5% NaOH in butanol ...	63.45	23.53	26.7
0.6% NaOH in 10% butanol + 90% white spirit ...	1.93	1.21	28.4
14.2% KOH in butanol ...	488.5	141.4	24.5
14.2% KOH in 10% butanol + 90% white spirit	4.955	3.52	29.2

Then, again, Gelmo and Suida (1905)<sup>49</sup> immersed wool in boiling 0.1% ethyl alcoholic potash for 45 min., and washed twice with alcohol and then with cold water; they obtained a material with a yellow colour but without apparent alteration in scale structure. Dyeing this treated wool in a neutral dyebath containing acid dyes showed no difference in shade between treated and untreated fabrics.

Thus, little or no fundamental or technical progress was made for about 40 years, till Hall and Wood took up the study from a different standpoint.

It was thought that, since non-aqueous solutions containing potential acid producers gave felt-resistant wool, the same effect should be obtainable with alkali-generators in non-aqueous media. The problem was how to achieve this.

A promising result was first obtained with an organic base dispersed in white spirit. Then a better result was obtained with metallic sodium dissolved in butanol diluted with white spirit, and a still better result with caustic soda dissolved in the same mixed medium. The immediate goal was to achieve felt-resistance and was not connected with any study of absorption of alkali. It was subsequently found that absorption studies

explained the superior result obtained when using ternary mixtures over binary mixtures (see Fig. 5-8, 13).

Fundamental data on solubility of alkali and water in non-aqueous liquids are given in Fig. 1-3.

Before the effects of some variables in the non-aqueous application of alkali to wool are discussed, the best general treatment for the production of non-felt wool is given.

The wool material, with or without a prescour (preferably with a light scour), is conditioned at

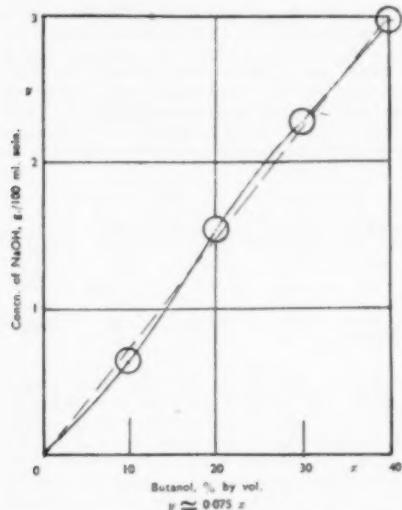


Fig. 1—Solubility of Caustic Soda in Butanol-White Spirit Mixtures at 18°C.

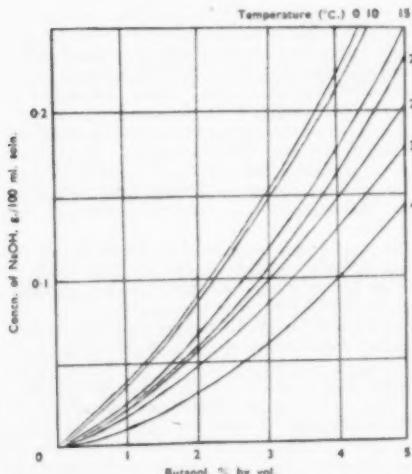


Fig. 2—Solubility of Caustic Soda in White Spirit containing 0-5% n-Butanol

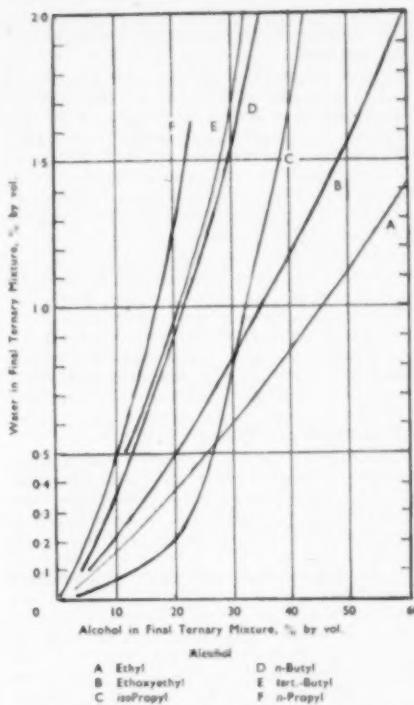


Fig. 3—Solubility of Water in Monohydric Alcohol-White Spirit Mixtures at 20°C.

about 70% R.H. and 70°F., so that it acquires a moisture content of about 15% (regain).

It is agitated with a ternary alkaline solution at 18-25°C. for 15-60 min. depending on the type of material undergoing treatment. The ratio of wool to liquor may be 5-15 : 1, and the liquor consists of 0.6 g. sodium hydroxide in a mixture of 10 c.c. n-butanol and 90 c.c. hydrocarbon (white spirit). The material is then well solvent-extracted (in a hydroextractor in the usual way), so that it retains not more than 2 gal. of reagent per 100 lb. of wool. The spent liquor is, of course, recovered for recausticising and reuse.

The solvent-extracted wool is quickly wetted in a suitably large volume of cold dilute sulphuric acid (2 Tw.), so that the alkali is immediately neutralised and there is no opportunity for dilute aqueous alkali to be found on the wool.

Thereafter the wool is rinsed, neutralised by treatment with a dilute solution of sodium carbonate, sodium bicarbonate, or a mixture of these, and finally soaped with warm liquor to remove all residual organic solvent. The material is dried to give a soft-handling non-felting product.

If the course of the reaction is followed under the microscope, it is found that the process is extremely mild and that the scale structure is

practically unchanged after the 1-hr. treatment, whilst the non-felting properties developed are extremely good.

It has been found that the wool fibre has suffered the very minimum of modification and that the action is almost entirely a surface one. *n*-Butanol ( $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ) and white spirit have molecules too large to enter the wool fibre, and this is confirmed by attempts to stretch wool in these liquids. Very little extension is obtained by stretching wool containing such molecules of more than three carbon atoms<sup>49</sup>.

The mechanism of the action is believed to consist in the very slight gelatinisation or incipient dissolution of the surface of the wool fibre. The protein on the surface is never actually dissolved, so that on acidification the minute layer which has been gelatinised is still keyed on to the protein substance. The effect is to cause the scale structure to be very slightly masked. This has been confirmed by microscopic examination and by electron-microscope studies.

This theory is further confirmed from another direction. Material which has been given the minimum standard treatment, and hence first-class shrink-resisting properties, can be rendered practically completely felting again by removal of this slightly surface-modified protein by treatment with suitable chemical hydrolytic and oxidising agents. Such solutions apparently remove the cementing substance, leaving the scales free to operate as before, thus again proving that the action is extremely mild and is confined to the surface. Where wool materials need not, or must not, be aftertreated in any way or where the anti-felting treatment is a final one, therefore, the

minimum standard treatment is all that is necessary, and this has been confirmed not only by laboratory-treated samples but also by works-production samples subsequently made up into garments and submitted to everyday use over a period of years. In other cases where the materials may be subjected to hydrolytic or oxidation processes, the minimum treatment is increased by the usual means, such as raising the temperature or lengthening the time of treatment at the ordinary temperature (Fig. 4).

As would be expected from theoretical considerations and the theoretical explanation of the process already given, chemical tests do not reveal any chemical change whatsoever, and the sulphur, nitrogen, and cystine contents and the alkali solubility remain unchanged even after the standard treatment has been prolonged for 2-4 hr.

Some values for the total sulphur (determined by Barratt's method<sup>50</sup>) and cystine contents are given in Table II.

TABLE II

Time of Treatment (hr.)	Total Sulphur (%)	Disulphide Bonds (%)	Thiol Groups (%)	Remaining Sulphur (%)
0	3.26	2.58	0.096	0.584
2½	3.31	2.53	0.09	0.69
3½	3.1	2.49	0.09	0.53
4½	3.3	2.42	0.09	0.76

Since the efficacy of the non-felting process depends upon the reaction with alkali at the surface of the wool fibre, it obviously depends on the rate and magnitude of the absorption of alkali, and this in turn will depend on the composition of the alkaline liquid presented to the wool, the duration of the action, the temperature, and the moisture content of the wool.

#### COMPOSITION OF THE ALKALI REAGENT

A very wide survey of the possible non-aqueous solutions of alkalis was made in the search for the ideal solution, and the first important observation was made that in general the ternary are much more effective than the binary non-aqueous reagents, particularly if the ternary solutions contain a preponderating amount of non-solvent for the alkali, viz. over 70% by volume. In addition they are cheaper. This effect was shown to be due to the greater absorption of alkali from such solutions: the non-polar substance is no mere diluent (Fig. 5-8).

The possible composition of an alkali reagent in a non-aqueous medium may be seen from a selected list of alkalis, solvents, and non-solvents given in the patent specifications<sup>25</sup>.

The superiority of the ternary over the binary reagents is shown in the diagrams, where the area shrinkage is plotted against the number of hand millings, which were so arranged as to give regular shrinkage steps for the untreated material.

The superiority of *n*-butanol-white spirit mixtures over all the rest is shown by these three series of curves (Fig. 5-7). The more the reaction of the alkali is confined to the surface of the wool fibres, the better the result obtained.

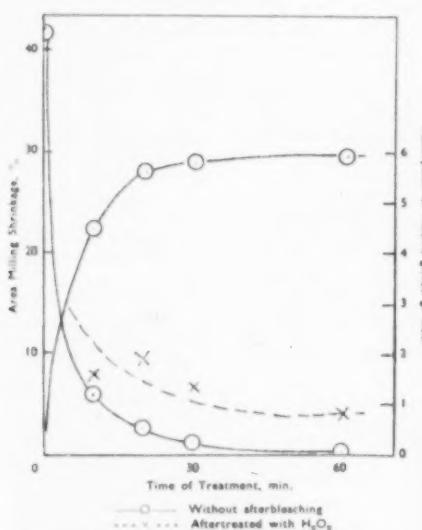


FIG. 4—Circular-knit Wool Fabric (C.K.F.) of Normal Moisture Content Treated at 25°C. in 10:1 Bath of Standard Solution (0.6% NaOH in 10% butanol plus 90% white spirit)

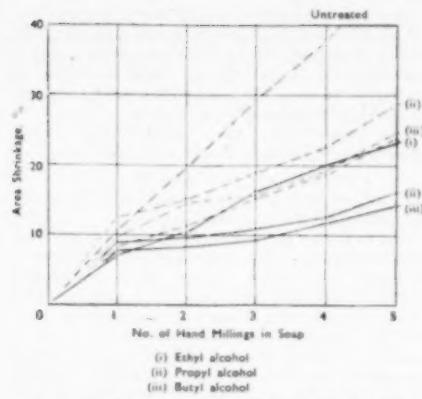


Fig. 5—Effect of Binary and Ternary Alcoholic Alkali Mixtures on Milling Shrinkage of C.K.F. (1 hr. at 18°C.)

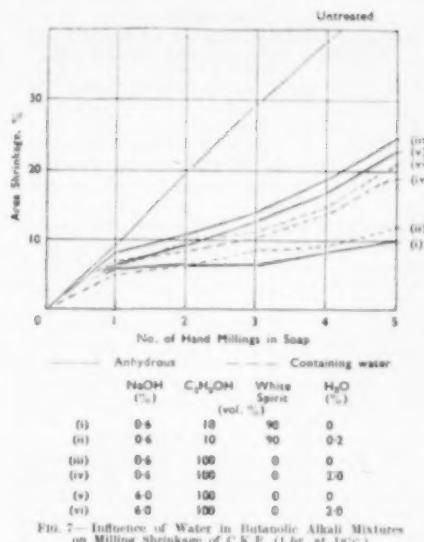


Fig. 7—Influence of Water in Butanolic Alkali Mixtures on Milling Shrinkage of C.K.F. (1 hr. at 18°C.)

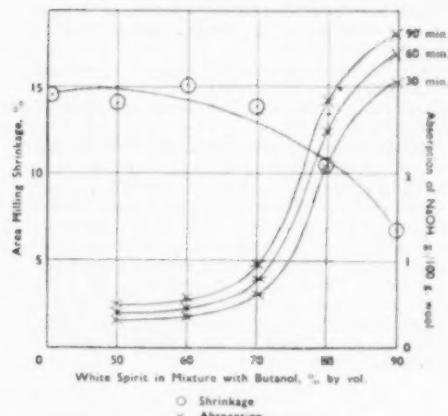


Fig. 6—Effect of Increase of Content of White Spirit on Alkali Absorption and Milling Shrinkage of C.K.F.

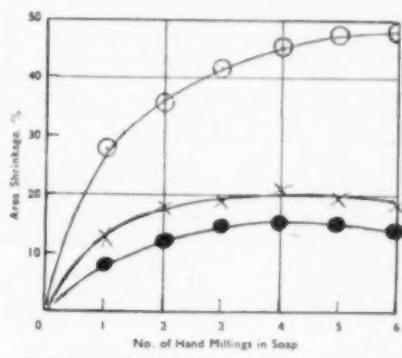


Fig. 8

The influence of the addition of small quantities of water to the reagents, both binary and ternary, is shown in Fig. 7. These curves demonstrate also that even 6% sodium hydroxide in butanol is not so effective as one-tenth of this concentration of caustic alkali dissolved in 10% butanol plus 90% white spirit. Best results and best control are obtained when the reagent is anhydrous.

#### THE TERNARY ALKALI REAGENT

During the course of the search for the most efficient ternary alkali reagent for application to

wool, binary solutions were made up and titrated with various non-solvents for the alkali to ascertain the limit of toleration of the non-solvent. Great variation was shown, and this also depended on the particular alkali used. Titration was continued till the alkali hydroxide began to separate out, and the end-point was checked by centrifuging the solution to find the point of instability. A few selected results are recorded in Table III.

Caustic potash is therefore generally not so useful or desirable as caustic soda, and is, of course, more expensive.

TABLE III  
Maximum Dilution Ratios of 15*N*-NaOH and  
15*N*-KOH in *n*-Butanol when diluted with  
Non-solvents

Non-solvent for Alkali	Temp. = 18°C. Vol. of 15 <i>N</i> butanolic alkali 10 c.c.					
	Sodium Hydroxide		Potassium Hydroxide		Vol. of Non- solvent (c.c.)	Dilu- tion Ratio
	Max. Vol. of Dilu- tion	Max. Ratio	Max. Vol. of Dilu- tion	Max. Ratio		
Benzene	30	3	12.6	1.3		
Toluene	35	3.5	13.7	1.4		
Xylene	40	4.0	17.5	1.8		
Carbon tetrachloride	1500	150	11.7	1.2		
Acetone	5	0.5	8.8	0.9		
Methyl ethyl ketone	2.5	0.3	7.2	0.7		
Dichloroethylene	400	40	4.4	0.4		
Trichloroethylene	7	0.7	4.4	0.4		
White spirit	90	9.0	40	4.0		

#### STABILITY OF THE REAGENTS

The dilution limits of a binary alkali reagent with a non-solvent obviously have an important bearing on the final choice of the particular non-solvent most desirable for large-scale use. Other factors include stability of both binary and ternary solutions, volatility (involving in this case recovery of solvents and flash-point), the effect of water added to the reagent, and the possible effect of the moisture in the wool entering the reagent.

When caustic alkali are dissolved in alcohols with or without the addition of a non-solvent, it might be expected that a reaction might occur and an equilibrium be established such as—



(M = Na or K). The equilibrium might be upset by the presence of a non-solvent. We have treated this problem in its simplest form with an immediate regard to large-scale working with the solutions in the following manner—

The stability of the binary solutions was observed for a month for both the common alkalis and mixtures of these in *n*-butanol. The interesting fact

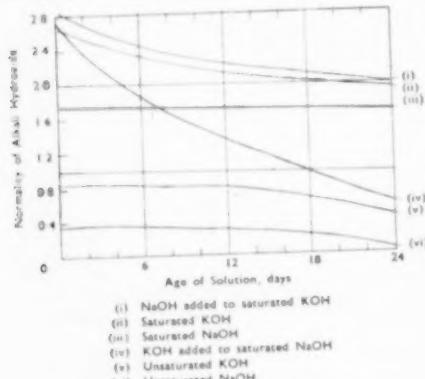


FIG. 9.—Stability of Caustic Soda and Caustic Potash dissolved in *n*-Butanol

emerged that saturated caustic soda in butanol was the most stable of all the mixtures tried (Fig. 9). This is demonstrated by recording the changes in concentration of the solutions with the lapse of time up to 26 days when the mixtures were allowed to stand in resistance-glass vessels.

The graphs show that, whatever the actual equilibrium state is in the possible reaction—



the system remains homogeneous and of reasonable stability for a month.

Observations on solutions of ternary mixtures prepared on the large scale (400 gal.) and kept in enclosed iron tanks showed that satisfactory stability was also obtained, indicating no progressive production of water in the sense of equation (ii), which would have resulted in removal of caustic soda from the system and become evident in a fall in concentration of caustic soda in the bulk of the non-aqueous solution. The composition of the solution was 0.6% sodium hydroxide in 10% (by volume) butanol plus 90% white spirit.

#### NON-SOLVENT CONTENT OF THE REAGEST

The effect on the caustic alkali absorption and milling shrinkage of the treated wool occasioned by the increase in the non-solvent content of the reagent is very greatly marked when about 70% or more of non-solvent is present in the mixture (Fig. 6).

On this remarkable effect, which occurs with all textile fibres, we have based our claim that the reagent is new for the treatment of textiles in general<sup>25a</sup>.

The difference between the effects of binary and ternary alkali reagents is specially marked where the binary mixture contains an alcohol of large dipole moment. Thus Cellosolve (ethoxyethyl alcohol) and butyl alcohol differ from each other only in that the Cellosolve has a non-hydroxylic oxygen atom in the molecule and is more polar. Anti-felting effects obtained with alkali in Cellosolve are not striking, but become exceedingly good when excess of white spirit is included in the liquid, i.e., when the polarity of the mixture is reduced (Table IV<sup>25a</sup>).

TABLE IV

Treatment	Area Milling Shrinkage (%)
Untreated	34
1.4% NaOH in Cellosolve	25
1.4% NaOH in 1 vol. Cellosolve + 22 vol. white spirit	1.6

#### MOISTURE CONTENT OF THE WOOL

It was found that the rate and magnitude of the absorption of alkali by wool from non-aqueous solvents depends to a large degree on the moisture content of the wool, at any rate for short periods of time reckoned in hours.

Fig. 10 shows the increasing absorption of alkali as the moisture in the wool is increased and also the effect on the milling shrinkage (area measurements). A large number of observations have been combined in the composite Fig. 10, which shows in

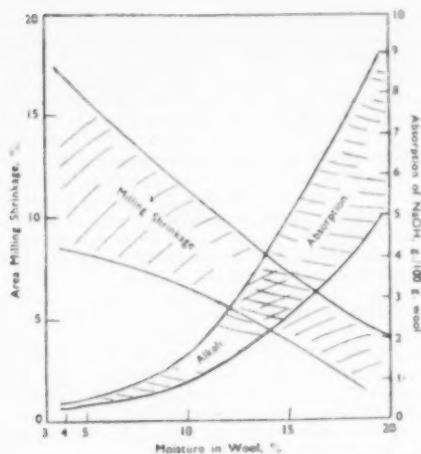


FIG. 10—Milling Shrinkage and Alkali Absorption of C.K.F. treated at 18°C. for 1 hr. in 9.4-12% Caustic Soda in 10% Butanol + 90% White Spirit (all the curves fell within the boundary regions marked)

a general way that, as the moisture in wool increases, the absorption of alkali increases and the felt-resistance also increases, i.e. the milling shrinkage decreases. Inspection of series of samples arising from this kind of study shows that the best results from all points of view are obtained when the moisture in the wool to be treated lies between 12% and 18%, regain, viz., the regain in a normal atmosphere of about 70% R.H.

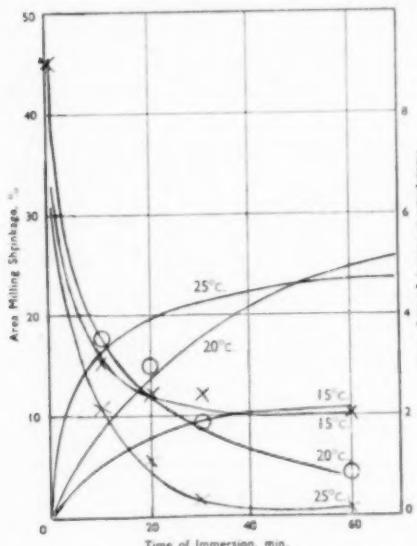


FIG. 11—C.K.F. containing 10% Moisture treated with 0.6% Caustic Soda in 10% Butanol + 90% White Spirit

#### TEMPERATURE OF TREATMENT

The rate of absorption of alkali and, therefore, the rate of reduction in felting power increase with rise of temperature, as might be expected (Fig. 11) illustrates this for wool containing 10% moisture.

An example of the rate of absorption of alkali and rate of reduction of felting power at 25°C. for wool with normal moisture is shown in Fig. 4, which illustrates that the first determines the second.

In general it has been found that the treatment of wool with a non-aqueous ternary alkali reagent, as disclosed in the T.B.I. patents<sup>2</sup>, should be carried out at approximately room temperature (generally 18-25°C.), since at lower temperatures, e.g. 10°C., the absorption of alkali is inconveniently slow, as is also the rate of chemical reaction, and at higher temperatures (above 30°C.) the absorption is conversely inconveniently rapid, and might lead to damage to the wool unless controlled. Further, at room temperatures the obvious difficulties, such as volatilisation of organic solvents, are avoided.

In connection with the temperature of treatment of wool and the stability of the ternary reagents, the latter possess an interesting property which has a bearing on the explanation of their superior efficiency as compared with the binary reagents. It was observed that on warming the standard ternary solution above 30°C. a very fine dispersion of caustic alkali was formed, and as the temperature was further raised the alkali was precipitated. On filtering such a solution at 65°C., 50% of the alkali was separated from a 0.6% caustic soda solution in 10% butanol in white spirit (cf. Fig. 2).

On cooling the heated solution from which caustic alkali has not been removed by filtration, the whole of it redissolves. It is quite clear that this phenomenon is directly related to the ease with which the wool takes up caustic alkali from such solutions.

Ternary solutions which on testing behave in this way give the best results when applied to conditioned wool for the production of a felt-resisting product.

#### EFFECTS OF PRETREATMENTS AND AFTERTREATMENTS

Any felt-resisting treatment given to wool should be applied at a stage at which it is as little changed as possible from its raw state. In general, if the wool has been damaged by aqueous alkali, it does not respond to the present treatments; whilst if it has been carbonised, it responds more quickly to the action of non-aqueous alkalis, and therefore a shorter period of time or a lower concentration of alkali suffices to produce a desired felt resistance.

#### ABSENCE OF THERMAL CHANGE DURING TREATMENT

The action of the alkali reagents on normally conditioned wool described in this paper is unaccompanied by any technically significant change of temperature. For example, 100 lb. of men's half-hose were immersed in 80 gal. of the standard ternary reagent at 18.0°C. in an earthenware pot. The temperature of the workroom was

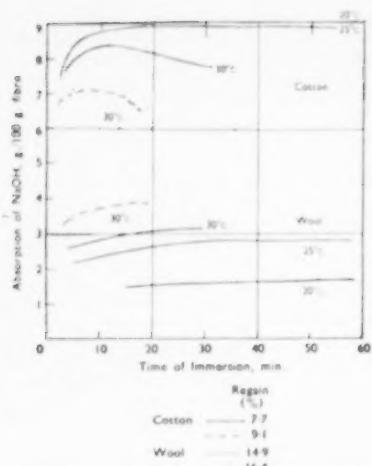


FIG. 12.—Partition of Caustic Soda from Butanol-White Spirit between Equal Masses of Wool and Cotton.

17.5°C., but the temperature measured by Beckmann thermometers at various points in the mass of material in the pot showed the same value of 18.0°C. during the course of 1 hr. This was checked many times during the processing of many batches of similar material.

This constitutes an important advantage in the processing of large quantities of goods, since a local evolution of heat is always detrimental to the uniformity of treatment.

TREATMENT OF WOOL-CELLULOSE MIXTURES

(a) Since cellulosic materials are not damaged by dilute or strong alkali solutions at ordinary temperatures, the application of non-aqueous alkali solutions would appear to be especially suitable for wool materials containing cellulosic and other fibres, and excellent results have been obtained on a large scale with mixtures of fibres.

In fibrous mixtures containing cellulose such as viscose rayon, cotton, linen, etc., it is obvious that both the cellulose and protein will take up the caustic alkali, and it is of interest to enquire into the relative amounts of alkali absorbed by the cellulose and the wool.

The partition of alkali between the cotton cellulose and wool protein was determined by immersing equal weights of the two materials in Hank form for the requisite time, taking them out, centrifuging off adhering liquor, and determining the alkali contained in the material.

Fig. 12 (Battye and Wood<sup>14</sup>) shows that the lower the temperature the greater is the amount of alkali taken out by a given weight of cellulose. The theoretical implications of this are interesting as fitting in with the known behaviour of the two materials. In spite of the lower moisture content of cotton cellulose, there appear to be more free reactive groups (hydroxyls) in cellulose than reactive groups in wool, which requires hydrolytic action before caustic alkali can be rapidly absorbed from the non-aqueous solution.

By means of varying states of conditioning the relative amounts of alkali absorbed by the two fibres may be controlled.

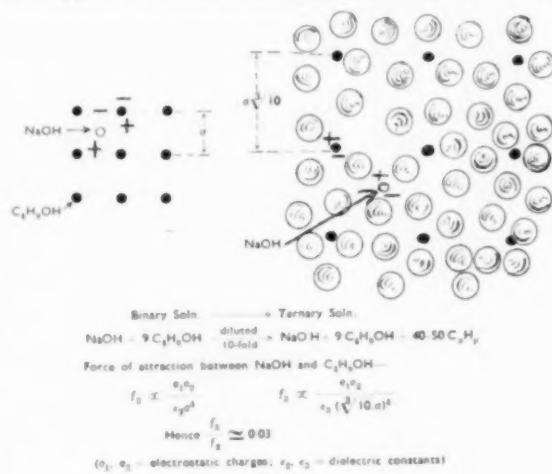
(b) From a theoretical point of view only, experiments were conducted to find the relative velocities of absorption of alkali when the two materials were not in competition for the alkali. It was again found that both viscose rayon and cotton absorb at 18°C. more alkali than does wool in 1 hr., the rates for cotton and rayon being approximately equal at both low and normal regains for both fibres.

Mixtures of wool with nylon or Terylene offer no difficulty in treatment: the nylon or Terylene takes up very little alkali, and its fluidity is unchanged, showing no adverse action on these synthetic fibres.

## THEORETICAL DISCUSSION

Particular attention has been focussed in this lecture on the theoretically unpredictable and surprising results which have been obtained in the application of non-aqueous alkali solutions to wool, where these solutions contain excess of a non-polar (insulating) substance.

It has been demonstrated that the non-polar non-solvent for the alkali acts not merely as a diluent (desirable as it is to have a cheap diluent) but that it enables the wood to capture more alkali from the polar alcohol present than it would otherwise do when the medium is purely polar (see Fig. 13).



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It seems quite obvious from a consideration of the polarity of the substances involved that the electric field between the caustic soda and the polar butanol, for example, is quite strong in the binary mixture containing 6% caustic soda. The molecular concentration of this solution would be about 1 NaOH : 9 C<sub>4</sub>H<sub>9</sub>OH. Now consider what happens when one volume of this solution is diluted to ten volumes with a hydrocarbon non-polar non-solvent. The average distance between the caustic soda molecules and the butanol molecules will be increased to, say, twice or thrice ( $\sqrt{10}$ ) its previous value, and on the inverse fourth-power law the electric field of force between the caustic soda and the butanol dipoles will be diminished to perhaps one-twentieth (5%) of its former value. But this is not all. In addition to this merely spatial separation of the two polar substances, they are now surrounded by a non-polar medium, thus further lessening to a great degree the electrical force between the two polar materials, just as a paraffin wax dielectric decreases the electric force and, therefore, increases the electrical capacity of a condenser. That this great reduction in electrical forces of attraction does occur and is qualitatively true is proven, since a rise of temperature causes this attractive force to be so much further weakened as to enable the caustic soda molecules to associate together and separate out from the solution.

Consider now what happens when wool (solid phase) is presented to the binary or to the ternary solution. In the first case the wool (1 part by weight) will be in contact with, say, 10 parts by volume of the polar solvent butanol and in competition with it for 0.6 part of caustic soda. It is clear that the electric field between the two latter is very strong. With the ternary solution, however, 1 part of wool is presented to 1 part by volume of butanol, 9 parts by volume of hydrocarbon, and 0.6 part of caustic soda; thus the competition for the latter is now between 1 part of wool and 1 part of butanol (of polarity weakened by being surrounded by 9 parts of hydrocarbon). It is quite easy to understand that in this second case the wool is able, if required, to exhaust the caustic soda completely from the bath. In simple language, if it is ensured that less butanol is taken up by the wool and that the molecules which are taken up by the wool are completely surrounded by hydrocarbon molecules, the caustic soda can get on with its task of attacking the protein surface.

The rôle of moisture in the reaction is a little difficult fully to comprehend, since even dry wool reacts in time with a fine dispersion of caustic alkali in a non-polar medium to give a felt-resisting product. Incipient gelatinisation, however, must be greatly assisted by small quantities of moisture, involving slight hydrolysis.

Although we are dealing with a five-component system, viz. protein-water-caustic soda-butanol-hydrocarbon, of quite a complex nature, the considerations given above suffice to explain in a qualitative manner the physical chemistry of the absorption phenomenon which has been experimentally observed.

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## Turkey Red Dyeing in Scotland Its Heyday and Decline

R. A. PEEL

*Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 19th February 1952.  
Mr. D. B. F. McAndrew in the chair*

Turkey Red, popularly associated with a vivid red of very great fastness which originated in Turkey or the East, was called at one time "Adrianoyle Red". We may assume from the scanty records available that the process began in the Near East and spread, through Arab influence, to India and the Far East. Scots have a better idea of the properties of Turkey Red than Englishmen, because Scotland introduced Turkey Red dyeing to these islands; she was successful in the very earliest stages of the process, and Turkey Red was dyed regularly in Scotland for 150 years.

The process was applied only to cotton, and, when first made public in Europe about the middle of the eighteenth century, it was found to be based on oiling, mordanting with alum, and dyeing by madder, the dyeing principle of which is Alizarin. (Turkey Red as we know it to-day is a fiery red colour lake formed on vegetable fibre by combination of a fatty acid with calcium aluminium alizarinate.)

Before Turkey Red dyeing could become successful the ground had to be prepared. We know from the records that yarn dyed with Turkey Red was manufactured in Glasgow in the year 1783. But we must look farther back to find how, by that time, the capital for investment was abundantly ready, the textile technique prepared, and labour confident and trained.

After the Union of Scotland and England in 1707, when Parliamentary Government was centred in London, the English monopoly of trade with the American Colonies and the West Indies was broken, and Scotland was free to trade. In 1707 Scotland owned 215 ships; in 1712 she owned 1123, owing to the alacrity with which the Scottish merchants and tradesmen seized their chance to trade freely overseas.

Important agricultural reforms were set afoot, flax growing expanding side by side with food production particularly in the East, the Carse of Stirling, and the Central Lowlands. In Dysart, Parish of Kirkcaldy, linen hand-loom weaving began about 1710, and developed into such a tremendous business throughout Fife, Angus, Perth, Renfrew, and Lanark that the British Linen Company was incorporated in 1746, imports of French cambrie were prohibited in 1748, and industrial Scotland was prosperous by 1780.

Mr. Creech, the publisher of Sir John Sinclair's *Statistical Account*, had some interesting comparisons to make, relating to Edinburgh—

In 1763 there was no such profession known as a Haberdasher. In 1783 the profession was nearly the most common in town. In 1763 the printed cottons manufactured amounted to 150,000 yards. In 1790 they amounted to 4,500,000 yards.

In the countryside the revolution was making itself felt. Away in the North the clearances had begun, sheep driving out the crofters. Crieff became a kind of clearing house for Highland labour finding its way to the industrial South. Many of the Highlanders who left home took up the business of bleaching, which was an open-air occupation. As early as 1728 Glasgow manufacturers sent their linen to be bleached on the banks of Loch Lomond and on the Leven. Dutch bleachers were employed at Cameron and Dalquhurn, which bleachfield, by 1715, was already one of the most famous in Scotland. Alan Ramsay wrote—

Come Meg, lets fa' to work upon this green,  
This shining day will bleach our linen clean,  
The water clear, the lift unclouded drew,  
Will mak them like a lily wet wi' dew.

A further development was the foundation of the first successful joint-stock cloth-printing company in Scotland, the Pollockshaws Printfield Company, founded in 1742 on land between the White Cart and the Auldhouse Burn by Archibald Ingram with John Glassford and other Glasgow merchants. Here gives an account of it—

Ingram bought the cotton wool, gave it out to spin, received it back as yarn for weft, and bought the linen warps. The process of weaving came under a manager. The dyestuffs it is true were bought at first hand. The cloth was sent to the field for whitening by an unskilled bleacher where it was afterwards put under the care of a master printer, whose skill was annually acquired by stealth from the working printers of London; where the manager was supposed to resort in the winter, and return to work in the Spring full of new information.

At first workmanship was so bad that Ingram nearly failed—

At last however, after a great waste of money abroad, and dyestuffs lost in experiments at home, he surmounted every difficulty and perseverance in due time met its just reward.

When the Pollockshaws Printfield Co. was dissolved in 1789, a number of textile works (including some devoted to Turkey Red dyeing) were built upon its land and fields, and there is one active and prominent dyehouse in operation there to-day.

Cotton, which was to replace linen almost entirely in the economy of the West of Scotland, was now in use, as has been seen, for the weft of some cloths, and cotton was needed for Turkey Red.

The *Statistical Account* reveals the remarkable spread of cotton spinning (and later weaving) throughout many parishes. In Kilsyth in 1793 beside a "York Copper Co." (note how these Yorkshiremen get about!) there was a Society of Weavers. The first cotton-spinning mill in Scotland was erected at Rothesay in 1778. And the village of Catrine in Ayrshire owes its existence to the cotton manufacturing started in 1787 by Mr.

Alexander of Ballochmyle and David Dale of Glasgow (the cotton works soon passed to James Finlay, and have been famous under that name ever since). At Strathaven in 1792 cotton manufacturing was firmly established. The girls worked at flowering muslin (i.e. tambouring—needlework with coloured threads), and the boys were weaving, too young, according to the parish minister—

... too early to that business which stunts their growth, occasions swelling about their legs and affects their morals by rendering them too soon independent of their parents.

David Dale, the father of the cotton manufacturing trade in Scotland, was a "maker" of Glasgow, far ahead of his time. Born at Stewarton in 1739 he was first a herd, then a peddler of yarns and cloth, and in 1763 he began in his own right at High Street, Glasgow, as a dealer in French yarns. From these small beginnings he extended his hold over all kinds of businesses, particularly in textile manufacturing. In 1783 he founded with Richard Arkwright and Robert Owen (Dale's son-in-law) the famous Lanark cotton mills, then the largest in Great Britain, and at about the same time combined with George Macintosh, the cudbear manufacturer, to set up Turkey Red dyeing. Macintosh, the same age as Dale, was a Highlander who, like Dale, had made his mark in Glasgow early in life.

To Dale, Macintosh, and many others who had already amassed power and wealth from Glasgow's looms and spinning frames, any textile process was of importance. In 1765 the French Government published an account of the Turkey Red dyeing process. The long standing ties of the Auld Alliance probably ensured that Scottish merchants had the news as soon as, if not before, their English counterparts. In Lancashire, it is true, Mr. John Wilson at Ainsworth, near Cockey Moor, had sent a young man out to Turkey in 1753 to learn the secret of Turkey Red. But Wilson was disappointed with the process, did not pursue it, and thus narrowly missed immortality.

Pursuing their interest in Turkey Red, Dale and Macintosh brought to Scotland one of the Frenchmen, skilled in the process, who were attracted by high wages to Great Britain.

Most descriptions of the start of Turkey Red dyeing in Scotland appear to be based on the *Statistical Account of Scotland, Volume 12—Barony Parish of Glasgow for 1794*, which reads—

The dyeing of Turkey Red on cotton though a very late discovery in this Kingdom was established in Glasgow earlier than in any part of Great Britain. In the year 1785 Mr. George Macintosh being in London fell in with M. Papillon a Turkey Red dyer from Rouen, carried him with him to Glasgow and in conjunction with Mr. David Dale built an extensive dyehouse at Dalmarnock in this parish upon the banks of the River Clyde, where cotton is dyed a real Turkey Red equal in beauty and solidity to East India colours. There is another dyehouse equally extensive lately erected for the same purpose in the neighbourhood of this one, also in the Barony parish under the management of M. Papillon who is now connected with another company. At both places the Turkey Red colours are now made in great perfection. By means of these establishments, the ingenious and industrious manufacturers of this place are enabled to make cotton palucate handkerchiefs equal in beauty and quality to any in the known world. And although the Messrs. Bouilles (one of whom is fixed

at Manchester) did obtain a premium from Parliament for the Turkey Red, the business was first established here, and specimens of manufactured palucates of a superior colour, it is said, were produced before a committee of the House of Commons (made by Mr. Macintosh who was the first to manufacture any here) while Mr. Bouille could only produce cuts of cotton yarn done by him. It is now computed that there are above 1500 looms employed in this branch of industry alone, in Glasgow and neighbourhood. The colour is so fast or fixed that when woven into brown cotton or linen yarn it resists and stands the whole process of bleaching and acquires more beauty and lustre by this trying operation; and when wrought in with bleached yarn requires 24 hours boiling in soap and ashes to reduce it to its vivid standard . . . Acids which destroy most other reds, in a moderate degree improve this. Making Turkey Red is a most intricate and troublesome process, requiring about 15 different operations in the common course of dyeing.

The name Bouille in this account is misspelt: Louis and Abraham Borell were French dyers, who, supported by the Manchester Chamber of Commerce, received in 1786 a £2500 reward from Parliament for revealing the secret of their Turkey Red dyeing process. M. Papillon had submitted samples of his own Turkey-Red-dyed yarn to a member of the Manchester Chamber in February 1785, but Borell had preceded him. However, by accident or design, George Macintosh secured the services of Papillon, and made rapid headway in Glasgow with his Turkey Red dyeing.

The following advertisement appeared in the *Glasgow Mercury* for 15th–22nd December 1785—

#### Dying

Dale and Macintosh have now got their Dyehouse finished and are just begun to dye cotton yarn Turkey Red for the Manufacturers, at 3s per lb. wt. The excellency of this colour is already known here, as it has been tried and found to stand the process of bleaching when woven along with green linen, or cotton yarn, without impairing, but rather increasing its beauty and lustre.

The yarn is received in by Mr. McDonald at Messrs. Macintosh and Murdoch's warehouse in Trongate, in quantities not less than 60 lb. at one time, from one person.

The manufacturers will please to loose the heaving bands and tie them slack in order to receive the colour equally; and put up the cotton in hanks from 4 to 6 ozs. each with a strong string. Each customer will get a particular mark to put on these strings which will be pleased to keep and use always for that purpose.

The *Statistical Account* mentions that Papillon had moved to another dyehouse—the following letter shows why—

George Macintosh Esq. Glasgow to Chas. Macintosh Esq. Glasgow. 18th January 1787.

Papillon has now left us entirely we could not manage his unhappy temper. I have made a great improvement in his process, I dye in twenty days what he took twenty five to do, and the colour better. We paid him his salary up to October, so as to be quite clear of him.

In 1787 it was intimated in the Glasgow Chamber of Commerce that the Government had purchased the secret of Turkey Red. The Chamber received a copy of the "Borell" method "for any members who may wish to see it". So it was obvious that the Papillon process might soon have a rival in the Borell method given to members of the Chamber. Yet the Borell method did not find root in Scotland. Papillon's process did, as is shown by this extract from the *Philosophical Magazine* (18, 43 (1804))—

*Account of Process followed by Mr. Pierre Jacques Papillon for dyeing Turkey Red*

It is now some years since M. Papillon established a dyehouse at Glasgow for dyeing cotton yarn Turkey Red or Adrianiople Red. In the year 1790 The Commissaries and Trustees for Manufactures in Scotland paid a premium to M. Papillon for communicating to the late Dr. Black then Professor of Chemistry Edinburgh a description of his process, on condition that it should not be divulged for a certain term of years. Those now being expired—the process is published.

*Papillon's Process*

[abridged]

*Step I or Cleaning Operation.* For 100 lbs of cotton take 100 lbs of Alicante barilla, 20 lbs of pearl ash, 100 lbs of quicklime. Mix the barilla with soft water in a deep tub having a small hole near its bottom which is to be stopped at first with a peg, but covered within by a cloth supported by two bricks, in order that the ashes may be hindered from either running through the hole or choking it, while the ley filters through it. Under this tub another is to be placed to receive the ley, and pure water is to be repeatedly passed through the first tub to form leys of different strength, which are to be kept until their strength has been examined. The strongest required for use must swim or float an egg, and is called the ley of six degrees of the French hydrometer or "pesse liquide".

*Step II. Barilla or Grey Steep.* Take a sufficient quantity (ten pails) of the strong barilla water in a tub, and dissolve or dilute in it two handfuls of sheep's dung. Then pour into it 2 quart bottles of oil of vitriol, 1 lb of gum arabic and 1 lb of sal ammoniac, both dissolved in a sufficient quantity of weak barilla water. Lastly 25 lbs of olive oil well mixed with weak barilla water. [This important last item omitted from the "Papillon" process given in Vol. I of *The Textile Colourist* for 1875.] Tramp, steep 24 hours, wring, dry, do this three times in all.

*Step III. The White Step.* Exactly as above, but omit the dung.

*Step IV. Galls Step.* Steep 24 hours in galls solution (milk warm).

*Step V. First Alum Step.* 25 lbs Roman Alum and strong barilla solution, 24 hours steep, dry.

*Step VI. Second Alum Step.* As last but after drying, steep six hours in river. Then wash and dry.

*Step VII. Drying Step.* Cotton yarn dyed 10 lbs at once, 2½ gallons ox-blood, 28 pails milk warm water, stir it well, then add 25 lbs of madder and stir all well together add yarn on stick, raise temperature over 1 hour to boil, boil 1 hour, wash and dry.

*Step VIII. The Fixing Step.* Alkali and dung, steep 6 hours, wring, and dry.

*Step IX. Brightening Step.* 10 lbs of White soap must be dissolved most carefully and completely in 18 pails of warm water. If any little bits of the soap remain undissolved they will make spots on the cotton add 4 pails of strong barilla water, sink the cotton, cover with cross stocks. Boil gently 2 hours, wash well and dry. The vessels required were four round coppers and a number of tubs of firwood, hooped with wood or copper. Not even a nail of iron.

There is a description by Dr. Black of a French hydrometer (later to be the "Twaddell") and also this note at the end—

Drying should be performed with particular care, and more perfectly than our driest weather is in general able to effect. It is done therefore in a room heated by a stove to a great degree.

Papillon's description is crude and simple, but Papillon was a dyer, and if the account was bluntly practical, it, at any rate, served its purpose. It must have given amusement to Messrs. Dale and Macintosh when published in 1804, by which time they would have improved it beyond recognition.

Why was the secret given to Dr. Black? Possibly because of his great reputation for chemical

research. He was often asked for advice by the Trustees and Commissioners for Manufactures, and George Macintosh also had asked his opinion on dyeing problems.

We have seen that Macintosh had dismissed Papillon, but it is interesting to follow the Frenchman's subsequent career. In June 1787 he advertised in the *Glasgow Mercury* that he had "set up a dyehouse" and had begun to "dye cotton yarn in Red, commonly called Turkey Red, of a colour superior to any that has been dyed in this country before except by himself" at "the French Street dyehouse at Rutherfordbridge near Glasgow". Later he was in partnership with Herbert Buchanan of Paisley, but this association was dissolved, and in September 1789 he was again in business on his own account and selling his wares "warranted full colour", through Messrs. Christie, Smith & Co. By March 1799, however, the *Glasgow Courier* carried this announcement—

*To be sold. By Private Bargain.*

The dwelling house, garden and grounds, with the whole of the Dyeworks belonging to M. Pierre Jacques Papillon, at Rutherfordbridge upon the River Clyde, in the neighbourhood of Glasgow. The premises have been laid out on the most convenient and approved plan for dyeworks, and may at a small expense be converted into a *Brewery* or *Distillery*. They possess many advantages for these or other employment that require a command of water . . .

However, the business does not appear to have been sold, for Papillon was still at Rutherfordbridge, dyeing cotton, in April of the same year, but no later advertisements appear in the Glasgow papers.

And what of Papillon? He had no further advertisements in the Glasgow papers of the early 1800s. He retired from business, and entrusted his money to his two sons, who lost it all, leaving him destitute. He died in poverty, but his widow received a pension, and a street in Bridgeton was named after him. It is French Street, but was once called Papillon Street. The original Dalmarnock Turkey Red works was situated in the area between French Street and the Clyde. Up to the 1840s this ground was mostly grass fields, where the cotton yarn and cloth were bleached and dyed Turkey Red in the summertime. Landressy Street also has Turkey Red origins. A French operative dyer who had worked for Papillon took the first house in a newly built street which was named after his home town, Landresy.

But if Papillon had come to grief, Dale and Macintosh had done well. In the year 1805, after a prosperous twenty years, Dale and Macintosh sold the Dalmarnock (Barrowfield) works to Henry Monteith of Carstairs, and a new era of Turkey Red dyeing in Scotland began.

We can almost say that the prominence of Turkey Red dyeing in Scotland is due largely to Rob Roy MacGregor. Rob's constant demands for a fee for the "protection" of the black cattle of James Monteith, a laird from near Aberfoyle, were refused. Three times the laird's stock was built up, only to be stolen, and so on his death his family were glad to move from the district. His grandson James became a master weaver in Glasgow, and it was his third son Henry, born in 1765, who made

Glasgow's Turkey Red dyeing of world-wide repute.

Henry Monteith's name, like those of his brothers, is in the Glasgow directory of 1789. He is listed as a muslin manufacturer at the address 3d. flat, Lightbody's land, south side, Bells Wynd. He was then only 24 years of age. In 1789 he passed successfully through a business crisis when, with cotton manufacturing in a depression, he lowered wages. The windows of his house were stoned, and in a scuffle with strikers in the street at Bridgeton his queue (pigtail) was cut off.

In 1802 he started the Barrowfield (now named Bridgeton) weaving factory of Henry Monteith, Bogle & Co., making bandana handkerchiefs, and rapidly established a high reputation in the weaving of these yarn-dyed articles—

which for bright and fast colours, and variety of design, raised the character of that branch of trade all over Europe. This branch of trade was confined to Glasgow—attempts on the continent were unsuccessful and remained so for over thirty years.

The same year he took over his brother James's weaving factory at Blantyre and started Turkey Red dyeing, which along with calico printing he developed at Blantyre and Barrowfield to a high state of perfection in dye and design on plicates, woven handkerchiefs (using Turkey Red yarn), and bandanas (Turkey Red with white and coloured discharges).

Henry Monteith introduced about 1813 discharge printing of the cloth by means of the Koechlin or Thomson process (BP 3654 (1813)), and soon after his fame became world-wide—

This is the celebrated patent for discharging the colour from the cloth by means of an acid and a solution of bleaching powder . . . . but recommends mixture of vinegar and tartaric acid, or a mixture of lime juice, crystallised citric acid, supersulphate of potash. The mixture thickened with starch and flour. The Turkey Red cloth hooked on frame and dipped in solution of bleaching powder.

In 1824 Ure could say—

The application of the chromate of lead on Turkey Red cloth forms a brilliant style of calico printing, now carried to high perfection at the establishment of Messrs. Monteith of Glasgow. Nitrate of lead is dissolved in liquid tartaric acid of a specific gravity about 1.250. This solution is thickened with gum and applied with the block to cloth previously dyed Turkey Red. Whenever the paste is dried, the cloth is slowly passed through an aqueous solution nearly saturated, of chloride of lime, kept at a temperature of about 100° F. in a stone trough. The tartaric acid disengaging the chlorine, discharges the colour of the Turkey Red at the points of application while the nitrate of lead, or rather perhaps the oxide of lead remains attached to the cloth. This is immediately washed and then passed through a solution of bichromate of potash by means of the padding machine. [This gave a bright yellow discharge.]

The Rt. Hon. Henry Monteith of Carstairs was Lord Provost of Glasgow in 1815-16 and 1819-20, and Member of Parliament in 1821 and 1831. Glasgow's population about the year 1820 was 150,000, and Monteith had an arduous task guiding the destiny of the growing city. His capable hand was there to alleviate distress during the cotton slump of 1816 at the close of the Napoleonic Wars, and also in 1820 when there was more unemployment. He died in 1848 aged 84, renowned, famous, his fortune built on Turkey Red. The Barrowfield

works continued to function until the retirement of Robert, son of Henry, in 1873, when they were demolished.

By now the Vale of Leven Turkey Red dyers were coming to the fore. There was increasing competition, and after a period of decay the firm of Henry Monteith was liquidated in 1904, the celebrated model works at Blantyre were condemned, and in 1925 the Livingstone Scottish National Memorial was built on the spot where Turkey Red had first become a big industry, and where David Livingstone had worked as a boy.

The Turkey Red process carried out at Monteith's in the 1850s must have been an adaptation of the Papillon process, i.e. emulsified olive oil. Red cloth would still be dried in the open air, and the process would be a seasonal one. The processes of the various works were kept very secret, and there was little fresh information published. For example, the *Encyclopaedia Britannica* in 1810 gave the Papillon process; there is no mention of the Borells, whom Parliament had so lavishly rewarded. In 1841 and 1855 the encyclopaedia published a description of the emulsified olive oil process. Cloth was left for fourteen days in the "soapy matter". It also reported that from Glasgow "the Turkey Red dye had gradually made its way to Lancashire".

John Thomson of Tradeston, in a letter published in 1816, described the perfection of Turkey Reds in Glasgow. He gave the process—ley boil, repeated immersions in olive oil emulsion with soda ash and sheep dung, warm washing in weak soda ash-olive oil emulsion immersions (no sheep dung), then gall and alum-madder dye-darkening steep in olive oil, and soda clearing.

Dr. T. Thomson, editor of the *Philosophical Magazine*, described the Scottish process as used in Glasgow in 1860—

A 16 step process with emulsified galipoli [olive] oil and soft sheep dung, plus soda ash, recommended as a 14 day steep, the longer the cloth is allowed to lie in the oil the better.

Improvements were not published, although Ure in 1824 recorded that the process could be modified and simplified—

A very eminent calico manufacturer in Scotland whom I consulted on the Turkey Red process assured me that the only essential mordants are oil and alumina, and that bright and fast reds, equal to any produced by the usual complicated process with sheep's dung, galls and blood, may be obtained without these articles.

The old-time orchid hunters observed the slogan "guard your stations" and kept all their activities secret; similarly, secrecy has always been an essential part of Turkey Red dyeing. It is obvious that great care and skill were needed to get perfect results from such a long and complicated operation. This knowledge would not be lightly given to others. It is difficult, then, to get more exact information about the firm of Monteith in the middle years of the nineteenth century, about the start of that well known Turkey Red dyeing firm in Cambuslang, T. P. Miller & Co., or of the start of Turkey Red dyeing in the Vale of Leven.

"The Vale", as it is fondly known to exiles all over the world, witnessed the rise and fall of Turkey Red in Scotland—

*The Vale*

Where cloth is printed, dyed and steamed,  
Bleached, tentered, in the water steamed,  
Starched, mangled, calendered and beamed,  
And folded very carefully.

The Reverend Mr. Gordon Stewart wrote of Bonhill in the year 1791—

The Leven is remarkable for the softness of its water, which fits in, in a peculiar manner for the purpose of bleaching. It is seldom or never muddy, as the rivers and burns from the Highland hills fall first into Loch Lomond where the mud they carry along with them subsides.

In 1792 there were 3 Printfields and 4 Bleach-fields in the Parish, . . . a Printfield started about 1768 . . . hand block handkerchiefs, then copper plate processes . . . Their eulogies for variety and fineness of colour are reckoned the first in Britain . . . For these two years past however, there have been violent disputes between the masters and the servants about the prices for the different pieces of work.

And the Rev. Alex. McAulay, writing on the Parish of Cardross—

In 1792 the Printfields of Dalquhurn and Cordale were by far the most considerable and extensive of any in Scotland. 876 persons were employed—of these 300 were girls (pincollers), and about 130 boys 8-15 years of age . . . the boys are in general healthy, active and lively, having nothing of that pale sickly look which so often marks those boys who are employed in the cotton spinning.

The bleaching was done in the open. Linen cloth, regularly sprinkled with pure water, was laid in the grass by narrow canals, with rows of beech hedges acting as wind-breaks between. The remnants of these beech hedges, now massive trees, still show where cloth was bleached in the old days.

William Stirling & Sons, the famous printers, purchased Cordale in 1770 and Dalquhurn in 1791. "Turkey Red dyeing was begun at Dalquhurn in 1816 but it was not until 1828 that the process began to be successful". The Turnbills came to the Vale, to Cordale, in 1770 with William Stirling, and later took a partnership for a short time in the firm. They established a chemical works at Millburn, which supplied the various bleach and print works of the neighbourhood, and were pioneers of the dyeing and printing trade in the Vale of Leven, being reputed to have been successful in dyeing Turkey Red at Croftengrea Works, Alexandria in 1827, i.e. before William Stirling & Sons.

Several other printworks (most of which were later to dye Turkey Red) were erected on both sides of the Leven from Renton to Jamestown. In 1841 Stirlings, followed by other firms, adopted the indoor drying system of Steiners, and largely dispensed with the Highlanders who had worked only in the summer months, returning to their homes in winter. This led to Irish immigrants settling in the Vale.

In 1859 the Dalquhurn works of Stirlings at Renton were dyeing 18 million yards of Turkey Red fabric and 800,000 lb. of Turkey Red yarn per annum. A brief outline of the process used at Dalquhurn in 1860 is given by Bremner. There were twenty-six operations of oiling, etc. before the madder dyebath.

The firm of John Orr Ewing & Co. of Alexandria, following the scientific trend of the times, engaged a qualified chemist for the first time in 1859, a

young man called John Christie, who was to leave his mark on Turkey Red, not that his name was to be personally as famous as Monteith's, but his knowledge of the process and his organising ability were later to create the largest Turkey Red organisation in the world.

At this time madder was the dye used on the aluminium-oil-calcium mordant. Madder is the root of a plant, *Rubia tinctorum*. The madder root (in its purified state called *Garancine*) was bulky, at least 100 lb. of madder being used for 100 lb. of yarn. In 1868 Alizarin was synthesised, and whereas in 1868 the amount of madder root produced was estimated at 70,000 tons a year, in a few years Alizarin almost completely replaced the natural material, and madder ceased to be grown.

From 1785 until the early 1870s the Scottish Turkey Red dyers had used Madder, Garancine, or Alizarin extracted from madder, imported from Holland, Turkey, and Japan. In 1863 Graebe and Liebermann, simultaneously with Perkin, synthesised Alizarin. The Badische Anilin- & Soda-Fabrik and Perkin had a monopoly of the British market, but Perkin sold out in 1874. It was not long after the discovery of Alizarin that Germany was offering it to the Vale dyers.

In 1873 Mr. John Christie, now a partner in the firm, was in Germany to visit Messrs. Friedrich Bayer & Co., apparently with the object of buying a substantial amount of Alizarin for John Orr Ewing & Co. He was accompanied by Mr. Roxburgh of Messrs. Archibald Orr Ewing & Co., Levenbank, and by Mr. Robert Tatlock, F.I.C., from whose diary the following extract is taken—

We were taken over the Alizarin factory and in the evening there was a dinner party at Mr. Rumpf's house which might have befitted a king . . . The fun was fast and furious and the champagne, Hock, Moselle and Strawberry Bowl flowed like water . . . I strongly suspected afterwards that one reason for this great demonstration of hospitality was that Mr. Christie and Mr. Roxburgh too, probably had orders in their pockets for large quantities of the new Alizarin dye amounting to several thousands of pounds value.

The German dye manufacturers used other methods besides lavish entertainment. After Perkin's retirement, they pursued the markets of the United Kingdom, which were largely Scottish. In 1877 Mr. Henderson of Henderson Hogg & Co., Glasgow, and Mr. Boulton, of Burt Boulton & Hayward, the successors of Perkin, and the main British manufacturers of Alizarin, were invited to visit Paris to meet members of the German cartel. For this meeting the German delegation had prepared a scheme for restriction. Production was to be reduced slightly below estimated world consumption. This meant that the British firms' share would amount to only one-half of their then production. Mr. Boulton, the British delegate, bluntly refused to do business on these terms, and the conference broke up.

In 1914 Mollwo Perkin, reviewing the dyestuffs position, referred to this period—

As a matter of fact, by combining together, the German manufacturers for a time practically killed the British Alizarin industry and had it not been for the Turkey Red Dyers' Association who combined to manufacture Alizarine, the trade would probably have entirely left the country.

The Turkey Red dyers of the Vale of Leven conceived the British Alizarin Co. in the interests of themselves and of Great Britain. With the active support of Lennox Lee of the Calico Printers Assocn. Ltd., the Marquis de Jaucourt of Messrs. Steiners of Accrington, and Mr. Boulton of Messrs. Burt Boulton & Hayward, distillers, of London, they formed the British Alizarin Company at Silvertown, London. The company was such a success that the Germans formed a cartel with it, and the markets of the world were apportioned, as was reported in the *Manchester Guardian* in October 1911—

The present Alizarin "ring" consists of the Farbenfabriken vorm. F. Bayer & Co. Elberfeld, the Farbwerke Hochst, the Badische Anilin und Soda Fabrik, Ludwigshafen, the British Alizarin Co., Silverton, and Wedekind & Co., Uerdingen. The British Alizarin Co., though a comparatively small concern, is well placed to meet competition for it is financially allied with the United Turkey Red Co. of Glasgow which uses enough of the dyestuff to keep a good part of the plant running. As the present price of 7d per pound for 20 per cent paste, Alizarin is certainly a profitable article for producers.

This cartel worked amicably and well, probably because the British side of it consisted of users interested in keeping the product at a reasonable price. It will be seen that Alizarin, so far as Great Britain was concerned, was Scottish-controlled.

The amalgamation of the Turkey Red dyeing firms in the Vale of Leven took place in 1898, when Turkey Red dyeing was in its heyday, the production in both yarn and cloth being colossal, for the markets of the world were practically theirs.

The concerns united were—John Orr Ewing & Co., Alexandria works, the largest, producing Turkey Red plain and dyed cloths, prints by blocks, by cylinder machines, by flat-press machines, by discharge presses, and Turkey Red yarns; the three works of Archibald Orr Ewing & Co.—the Levenbank works producing plain-dyed and a great variety of printed Turkey Red and Alizarin prints, the Dillichip works producing solely Turkey Red and Aniline Black yarns, and the Milton works producing only Turkey Red yarn; the two works of William Stirling & Sons, Renton, viz. Dalquhurn, plain Turkey Red, and Cordale, Turkey Red and Alizarin prints; the small chemical works, Millburn, producing acetic acid, calcium and aluminium acetates, methyl alcohol, and acetone; all the above in the Vale of Leven, together with Alexander Reid & Sons of Milngavie, dyeing Alizarin Red and fancy colours on yarn. Together these firms became the United Turkey Red Co. Ltd. (U.T.R.).

Though this association made all these concerns one in respect of finance, yet each preserved its identity in trading. Indeed, the products of the respective branches appeared on the foreign markets under the old works names, as if no amalgamation had taken place. This policy was necessary, for in those days the products of the Vale of Leven Turkey Red concerns were sold in the foreign markets of India, China, the Philippines, Africa, South America, and the South Sea Islands under trade marks in the form of gaudy lithographed tickets.

The dyeing concerns provided the cloth either dyed or printed, and also the yarns, and consigned these to large merchant houses abroad, who, in turn, sold them to the native dealers or auctioned them. Always the products were known by the lithographed tickets or marks, sometimes as many as six tickets being stuck on a 30-yard piece. Goods were nearly always consigned for sale; i.e. they went to the godowns of the merchants for sale, and were seldom sold or indented to merchants in this country.

When Turkey Red dyeing was originally begun in the Vale, i.e. about 1816, and for long afterwards, it would be by the "emulsion process" based on olive oil, *huile tourante*, slightly acid or rancid, emulsified with pearl ash, the fabric being actually dried in the open air in the summer months after the application of the emulsified oil liquors, so that it was not a process that could be practised all the year round, not at any rate until heated stoves were installed.

In 1898 two processes were used at the works of John Orr Ewing—(1) the Steiner, called the "old" process and (2) the sulphonated olive oil process. The former was not applicable to yarn, and further it was considered dangerous at the stoving stage, many a building having been burned by its use. The outline of the Steiner process is as follows—The cloth was padded in the natural, i.e. untreated, olive oil, and then stoved in a heated chamber for a few hours up to 180°F. After this first stoving the cloth was treated, continuously and in open width, a number of times with weak potassium carbonate solution, stoving to 180°F. after each treatment; the cloth was then mordanted with a basic solution of potash alum and aluminium acetate, and left wet in the mordant for 36 hr., washed well after mordanting, avoiding much pressure on the face of the cloth, and then dyed with Alizarin, blood albumen, sumac, and chalk to a temperature not exceeding 200°F. in 2½ hr., the dyebath being started at about 80°F. After dyeing, the cloth is cleared twice with soap and a little tin crystals in enclosed copper pots at a pressure of 8 lb./sq. in., washed, and dried in sheds over spars without heat.

The "sulphonated olive oil emulsion process" was the most important in the Vale of Leven during the last fifty years of the industry, and was applied to both cloth and yarn. An outline follows—Cloth was prepared for the process by boiling in caustic soda for 10 hr. at 20 lb./sq. in. pressure. Dark "husk" cloth was sometimes chemicked with ½ Tw. sodium hypochlorite made neutral with sodium carbonate. All water pipes in the bleaching croft were of pure copper to guard against iron being deposited on the cloth during washing, for the merest trace in the oil process would show when the dyebox stage was reached. The oil used in this process was a mixture, roughly 5 parts of sulphonated olive and 1 part of sulphonated castor (ammonia olein 50%) emulsified in a solution of potassium carbonate. The cloth was subjected to four treatments with the oil liquor, being stoved to 180°F. after each; the yarn to four treatments, and for the finest non-smearing Turkey Red such as used in the

Irish heavy-friction calenders, to five treatments, the last being with weak potassium carbonate solution. After the oil treatment the cloth or yarn was mordanted with a mixture of basic potash alum and red liquor, extra precautions being taken to secure an iron-free mordant. Cloth and yarn were both steeped in the mordant solution for 36 hr., then washed, dyed, cleared under pressure (twice) with soap and tin crystals, and finally air-dried.

Who at this time (1898) were engaged in the production of Turkey Red in Great Britain? Outside the dyers in the Vale of Leven, i.e. the U.T.R., there was only one major firm in Scotland, T. P. Miller & Co., Cambuslang, which produced Turkey Reds used only by the Irish linen finishers. There was a smaller firm, J. & W. Campbell of Pollockshaws, founded in 1851 and dyeing hanks (cotton yarn) by the short (sulphonated castor oil) Turkey Red process. As such they could not be classed with the much faster Red production in the "Vale". In England Messrs. Steiner produced an excellent plain-dyed Turkey Red on cloth and also Turkey Red cylinder print discharges; the British Cotton & Wool Dyers Assocn. Ltd. at Baxenden in England produced a Turkey Red on yarn for the home trade, a colour nearer to the classification of Alizarin Red. In Germany there existed one or two excellent Turkey Red yarn dyers, for instance Bemberg, but whilst now and then their products were seen in the British markets, they were never met in foreign ones. Besides, neither the Swiss nor the Dutch concerns competed in the Vale markets, and that explains why Vale dyers had practically the world market to themselves. Neither the Bradford Dyers Assocn. Ltd. nor the Calico Printers Assocn. Ltd. produced Turkey Red, though the latter produced plenty of Alizarin Red prints.

The monopolistic nature of the Turkey Red industry can easily be explained. To begin with, it was a highly specialised craft that had been slowly evolved over generations by a host of contributors, and practically no up-to-date information about it was to be found in textbooks or in lectures on dyeing. None of the technicians holding important posts in the U.T.R. was allowed to deliver lectures on the process or to write articles for publication in scientific or trade journals. No representative of a dye or chemical firm was allowed to enter the works, and so little leaked out that it is not surprising that books on dyeing had little to tell aspiring entrants to the industry. Of course, Hummel and others who had worked in the industry were able to print lengthy accounts of the processes, as had Berthollet and Papillon before them. But these accounts did not reveal "know-how" nor new developments.

Processes were so carefully guarded in those days that few inside the works themselves knew anything more than the broad outline or general method. If a very special and new method was on trial, it was called a "secret work" and kept a secret.

Moreover, the Turkey Red plants in the Vale were huge affairs as plants go in the general dyeing trade, and as costly as they were huge, only a world-wide off-take being capable of supporting

them. Further, they covered large acreages and consumed, daily, millions of gallons of water, so that they could not be placed in urban areas, where land was dear and water was metered. Again, the water used must not be contaminated in any way, and neither too hard nor too soft. Lastly, in the heyday of Turkey Red, the production was sold in the large markets abroad, under trade marks, and, when a first-class business is built up on trade marks, it is not easy for a new and unknown mark to make headway.

The Turkey Reds produced in the Vale, and by Millers' of Cambuslang, were fast and vivid reds, traditional Turkey Reds. Opinion then as to what constituted a Turkey Red was fully in accord with tradition, viz. a colour lake formed with an oxidised fatty acid such as oxyoleic or trioxyoleic acid, in combination with aluminium oxide, Alizarin, and calcium. It is very important to note that the alumina of the colour lake must be entirely fixed by the fat on the fibre, which means that the fat must be present in sufficient amount to do this. A "Red" made up of fat, alumina, and Alizarin, where the fat was not sufficient to fix the full amount of alumina necessary for the lake, and where the fixation had to be completed by the use of such agents as "binarseniate of soda" ( $\text{NaH}_2\text{AsO}_4\cdot\text{H}_2\text{O}$ ) and chalk, sodium phosphate, and even sodium silicate, was excluded from the Turkey Red class, and was called "Alizarin Red".

The U.T.R. produced both in large quantities, but never on any account were the two mixed. Indeed, it would have been futile to mix them, for the fastness of the two was entirely different. A properly processed Turkey Red will stand pressure boiling with sodium carbonate followed by a bleaching treatment with sodium hypochlorite provided this latter is kept on the alkaline side with soda ash. It will also stand a weak caustic soda boil, but users were discouraged from applying this. It will stand soap boiling. It will stand British daylight and sunshine for over 2½ years perfectly. On yarn it should be soft to the handle, free to wind and weave; and in the highest class of red it should not rub off when a hank of it is wrapped with white crash linen, and the linen is rubbed back and forward with all the force one can exercise. This latter red was considered the high-water mark of the industry. In other words, it was the ordinary Turkey Red given the extra "non-smearing" quality by extra processing at the oiling stage.

Turkey Red cannot be produced by a printing process, but there was a very large printing business in discharging cloth-dyed Turkey Red. The discharging was carried out by the old classic method, by printing citric acid and then passing the cloth through a vat of calcium hypochlorite. Colours other than white were obtained by incorporating with the citric-acid-printed impression—Prussian Blue for blues, lead salts followed by bichrome for yellows, a combination of the blue and yellow for greens, and logwood for blacks.

Turkey Red five-colour discharges in elaborate floral designs, on good cotton twill cloths, were very fashionable 30–50 years ago, particularly as bed-spread chintzes. Besides cylinder printing and

block printing, there were discharges by a special process known as "lead plate". This was a discontinuous process, the red cloth being fitted between two large pure lead plates with the patterns (usually large floral designs) cut deeply in intaglio, to form cavities; the lead plates were screwed face to face with great pressure, and hypochlorite of lime, slightly on the acid side, was circulated in the cavities forming the pattern, by means of a pump.

In this discharge by lead plate usually only two colours were produced, white and yellow, the yellow being merely the white treated with a lead salt and bichrome. All lead-plate production went to a group of islands in the South Pacific. One other discharge process was by flat press, a discontinuous process also, where the cloth was pressed against an engraved copper plate of large dimensions. All these discharge processes produced their unique features; hence their existence side by side.

A great quantity of Alizarin was at one time used by calico printers, and the reds produced almost invariably contained fatty matter, aluminium oxide, and Alizarin; but the mordant (the alumina) in the lake was not fixed solely by the fatty matter. Indeed it was fixed only in small proportions by the fat, so none of these printers' reds would be classed as Turkey Red.

In the early 1900s, when economies were forced on the U.T.R. works, the J.F. process of dyeing Turkey Red was invented and developed by John F. Christie, son of John Christie, Chairman of the U.T.R. This was a cheaper and shorter dyeing process than the "old" process, and was carried out as follows—

Pad the cloth in the sulphonated castor solution (1.53 gal. 50% soda oil, 0.17 gal. 50% ammonia oil, 9½ fl.oz. sodium stannate 40° Tw.—total 1.7 gal. per 100 lb. cotton); mangle with floating nip, and top bowl lapped carefully with thin flannel, and from the mangle cloth run wet into a "cave" (which when full can be covered to exclude air and prevent local evaporation); leave in the "cave" for at least 6 hr., run through the oil solution again, and dry over cans.

The stitching of one lump of cloth to another must be perfectly level, and without puckers, or otherwise creases will be caused that will show in the final result. Drying cylinders must be run at a speed to give dry seams, otherwise damp patches will occur, i.e. the oil layers will be disturbed in the steaming process. After drying, the cloth is rolled loosely, lump by lump, and wrapped with sheets to keep as warm as possible for its entry into the steaming chamber. This is like a large Lancashire boiler with internal wooden framework, and this framework is completely covered by hessian sheets which have been boiled with water under slight pressure to remove any sizing matter used in the spinning process. The cloth is put into the steaming chamber in the loose-rolled lump form, and covered with sheets, and the hot-air appliance on the steamer is turned on for 30 min., so that the cloth will not condense the steam, which of course would be fatal. The steam must not be superheated, for that would char the oil, especially the ammonia part of it. If oil is browned in the steaming process to any extent, the resultant colours will

be correspondingly dull. The cloth is steamed for 90 min. at 15 lb./sq. in. pressure, and when taken out of the steamer is built in criss-cross fashion to allow steam to evaporate without condensing (this must be done in a warm room). As soon as possible, cloth is dried again over copper cylinders and sent for mordanting.

The mordanting in Turkey Red is just as important as the laying of a proper oil foundation, and the aluminium oxide must come down in as fine physical state as possible. One can mordant well oiled cloth with a number of aluminium salts, e.g. potash and soda alums, aluminium sulphate, acetate, formate, etc., but the resulting colours are widely different. No one salt is equal to a blend.

Further, an aluminium salt for the Turkey Red process must be made alkaline by the addition of soda ash. The mordant used was—

847 lb.	Potash alum	<i>All iron free</i>
84 lb.	Soda ash	
70 gal.	Aluminium acetate (15° Tw.)	

$\left\{ \begin{array}{l} 1028 \text{ gal. of } 10^{\circ} \text{ Tw.} \\ 1.2\% \text{ Al}_2\text{O}_3 \\ \text{acetate (15° Tw.)} \end{array} \right.$

Mordanting takes place in a padding machine, brass bowls lapped with flannel. Nip 13 gal./100 lb. Mordant heated to 120°F. with closed coil. Cloth run through this machine and into large "caves", where it lies well covered for 36 hr., after which it is washed in a range of spiral washing machines (no nip) and finally run up an inclined washing trough. Then the washed cloth is very lightly squeezed. These precautions in washing are necessary, because at this stage the alumina-oil precipitate is "soft" and likely to be disturbed by pressure. The next step is to open the cloth to full width. This opening is done by hand, as it was found that mechanical openers disturbed the oil-alumina layer. Cloth is now dyed in a 300-gal. winch, allowed to run a few minutes in 1 gal. of bullock's blood (or 1.36 lb. of blood albumen) and then 4%, chalk with ½ lb. sumach. Run for 5 min., add Alizarin, and run a further 5 min. Turn on steam, and slowly raise temperature to 160°F. in 110 min. Run for 10 min. at 160°F., and draw cloth out. It is now washed and squeezed without pressure, for the lake is still "soft". The cloth is opened by hand to full width, squeezed in a wooden bowl mangle to a definite water content, and run through another with a much slackener nip. This last mangle contains soda sulphonated oil (1 : 1), the lift being only the difference between the expression of the two manglings. From the second mangle, cloth is dried over copper cans, and then steamed for 3½ hr. at 20 lb./sq. in. pressure, avoiding superheated steam. After steaming the lake is now firm, and the red cloth is run through very weak caustic soda in a spiral machine, washed, and cleared twice in copper pots, under slight pressure, first with—

5.3%	Cotton oil soap
2.7%	White tallow soap
0.33%	Tin crystals

and then with—

2%	Cotton oil soap
1%	White tallow soap
0.165%	Tin crystals

Then the cloth is washed well, and dried over spars at air temperature in large sheds.

On cloth the J.F. Turkey Red process proved to be a great success, and from 1908 its production gradually grew, until it assumed gigantic proportions, both as a plain dye and as a ground for discharge printing. While it grew the sulphonated olive oil process correspondingly diminished. On yarn the J.F. was thoroughly tried out, but was unsuccessful. On cloth it was sufficient to satisfy the demands of domestic wear, viz. good fastness to light, washing with soap, and marking off. On yarn the demands were much more severe; it had, of course, to be fast to light and soap washing, but in addition it had to wind and weave well, and be dyed through to an extent unknown in cloth, so that if made into chenille goods, or towel headings, no white would show.

In 1898, besides Turkey Red, both Benzo Red and Paranitraniline were produced, the latter in nothing like the volume it was to reach a few years later, when Para Red was greatly improved in shade and in non-smearing property. It may seem strange that these two dyes, especially the Benzo, so greatly inferior to Turkey Red in fastness, could exist alongside the latter. But such is the case, and even when Paranitraniline was produced in huge quantities for the millions of China, its prosperity did not encroach on the Turkey Red sphere.

Benzo Red is on the lowest rung of the fastness ladder, and was destroyed in a few hours by the Indian sun, but then it was the only red at that time that the very poor people could afford, so this colour had a sphere of use based on cheapness.

The same applied to the faster Para Red, with a sphere of its own and a rather richer clientele. Para Red, after two days in the Indian sun, was reduced to something like a brick shade. Turkey Red was much faster in every way than these two dyes, but it was much dearer and had wealthier customers. So these three dyes existed side by side, at three different prices and supported by different consumers.

Turkey Reds were dyed in India, of course, but the shade was more of a chestnut or Indian red, not so bright as the Vale Turkey Red. This arrangement of production, with Turkey Red a long way ahead of its rivals in fastness, might have lasted indefinitely, but for the loss of Indian markets due to the development of cotton spinning, weaving, and dyeing in India, and the imposition of import duties on yarns and cloths.

Another development was the rise of the Naphthol Reds. These were tested carefully by the U.T.R. when first produced in 1914, and eventually dyed as trial bulk orders for India and China. They were given new trade marks, but were not well received at first. It was quite evident, however, that this new class of dye, which was so much faster than Para, so brilliant in shade, and so easy to apply, would in the long run disturb Turkey Red. The First World War prevented the rapid development of these azoics, and, for several years after Turkey Red was in good vigour, but the new Naphthols with fastness equal to Turkey Red in domestic wear had by 1936 killed Turkey Red both at home and abroad. The British Alizarin business was seriously depressed when Turkey Red

dyeing began to decline. As its principal product was less in demand, the intention to expand from London to Trafford Park was abandoned, and the company was taken over by the British Dyestuffs Corp. (now ICI).

How did all these vast processes, in which millions of gallons of water were used, affect the River Leven? How did the works and industry affect the amenities of the Vale? Early in the eighteenth century Tobias Smollett, born in the Vale and its most famous author, had written in his *Ode to Leven Water*—

No torrents stain thy limpid source,  
No rocks impede thy dimpling course,  
That sweetly warbles o'er its bed,  
With white, round polished pebbles spread.

In 1803 Dorothy Wordsworth wrote in her famous journal, *Recollections of a Tour made in Scotland*—

The Vale is populous, but looks as if it were not inhabited by cultivators of the earth, the houses are chiefly of stone, often in rows by the river-side, they stand pleasantly, but have a tridish look, as if they might have been offsets from Glasgow. We saw many bleach-yards, but no other symptom of a manufactory, except something in the houses which was not rural, and a want of independent comforts.

A later writer was definite in his dislike and condemnation—

One may briefly picture then this somewhat drab southern approach to Loch Lomond. Leaving behind the broad estuary of the Clyde one turns northwards into the valley of the Leven. A valley this, whose bleak moorland sky-lines gain nothing of cheerfulness from the smoke which drifts from the factory chimneys and obscures the sordid dwellings along the filthy river, a valley in which at Balloch, throughout the summer, concentrate restless and noisy city crowds . . . Not a pretty picture, certainly, but one in striking contrast to that other, which lies a bare half-mile to the north, where a vast panorama of mountain and loch opens itself suddenly to the delighted gaze of the visitor.

Lamond, artist and fisherman, was rather hard on the Vale. There is some truth in his strictures, of course. But few industrial areas are so fortunate in their situation: this one is on the edge of Paradise. Compare the Leven with the Clyde at Bridgeton, or with the Cart—the once White Cart—and then think how bonny the Leven yet is. Though industry has apparently hemmed it in, it still runs clean, remarkably free from pollution, and when it is polluted (as has been known to happen even in these days), there is very soon a hue and cry about it.

The products of the Scottish Turkey Red dyers were exported all over the world. The patterns shown in the old books reveal a craftsmanship, artistry, and patience worthy of a connoisseur's attention.

There is a profound and logical sense of design and working rhythm in the industrial Lowlands, and in the West of Scotland particularly. If Turkey Red is no more, the printing and dyeing industry still survives. If these trades do not employ the same numbers as when Turkey Red was in its heyday, other occupations have been found. The nimble fingers, the sense of pattern, the eye for detail and symmetry are making new industries in the formerly depressed areas of the West. The Turkey Red industry was a craft, and persisted in its tradition of craftsmanship in spite

of mass production. The craft qualities which were later to build a ship like the *Queen Mary* were first developed in the bleachfields and early textile industries of the Clyde Valley.

The Turkey Red process also made a few millionaires: it satisfied the land hunger of many a Scottish businessman, by placing him in the landed gentry. It flourished in good soil. Andrew Brown chose an apt quotation from Goldsmith for the flyleaf of his book *The History of Glasgow*, published in 1795—

Industrious habits in each bosom reign,  
And industry begets a love of gain,  
There much lov'd wealth imparts,  
Convenience, plenty, elegance and arts.

It was said during the great depression of 1933 that the Vale was down and out. But, "in the Vale there are elements which defy destruction" says Ian Lees. The leaders of its main industry were among the first in Scotland to reorganise and rationalise, and, when Turkey Red ceased, other dyeing methods on other fibres were prepared ready to take its place.

There is a poignancy in saying farewell to a cherished time or epoch—the old days are always the best days in memory's eye. We look back and remember with pride those who built up Turkey Red in Scotland.

And we, who carry on the textile and colouring traditions, can think on the words of Tobias Smollett—

And Ancient faith that knows no guile,  
And industry unbrown'd with toil,  
And hearts resolved and hands prepared,  
The blessings they enjoy to guard.

\* \* \*

A large number of dyed and printed Turkey Red fabrics were on view at the lecture.

\* \* \*

The author wishes to thank Messrs. The United Turkey Red Co. Ltd. for permission to publish this paper; Mr. J. F. Christie for advice and encouragement during its preparation; the Librarians of the Mitchell Library, Glasgow, the University Library, Glasgow, and the Templeton Library, Helensburgh; the Editor of the *Dyer*; Mr. C. O. Clark for the loan of books and references; and many individuals who through correspondence and discussion have contributed to this paper.

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## COMMUNICATIONS

## Combination of Wool with Acids

BERTIL OLOFSSON

1. Titrations of wool with mixtures of hydrochloric acid and sulphuric acid in different total and relative concentrations have been performed, and the absorption of hydrogen, chloride, and sulphate ions has been determined.

2. The experimental results are compared with results calculated on the basis of the Gilbert-Rideal, Donnan, and ion-exchange theories.

3. Statistical analysis proves the applicability of the Gilbert-Rideal theory. The Donnan theory is not applicable, as there are specific thermodynamic differences between fibre and solution phases. The ion-exchange theory is more general, including the other two theories.

4. A theoretical study of the attachment of the sulphate ion at one or two positive sites in the fibre has been made. Experimental values are not conclusive, but make it possible that there is either a one-site attachment or a distribution between one and two sites.

## INTRODUCTION

In a previous communication<sup>1</sup> was reported a series of investigations on the quantitative aspects of the absorption of sulphate and chloride ions on wool in the presence of sodium ions. To verify the conclusions reached and to obtain a sharper quantitative interpretation of the phenomena, a new series of experiments has been made using hydrochloric and sulphuric acids, in absence of sodium ions, in mixtures of different composition and different acid strength.

## SYMBOLS

The symbols used are the same as in the earlier paper<sup>1</sup>.

$a$	Concentration of ionic groups on fibre in volume $v$
$A_X$	Quantity of $X$ absorbed in millimoles per gram of dry wool
$\theta_X$	$A_X / A_{X \text{ max}}$
$\theta'$	Number of $\text{COOH}$ groups on fibre
$\theta'$	Number of $\text{COOH} + \text{COO}^-$ groups on fibre
$c_X$ or $[X]$	Concentration of $X$ in gram-molecules per litre
$a_X$ or $[X]$	Activity of $X$
$f_X$	Activity coefficient of $X$
$\mu_X$	Chemical potential of $X$
$\mu_X^*$	Chemical potential of $X$ in standard state (at given pressure and temperature)
$\mu_H$	$\mu_{\text{H}}^*$ (fibre) — $\mu_{\text{H}}^*$ (soln.)
$V$	Electrostatic potential of fibre
$z$	Ionic valency
$F$	Faraday
$R$	Molar gas constant
$T$	Absolute temperature
$k$	Donnan quotient = $e^{VFR/T}$
$v$	Volume of internal solution per gram of dry wool
$I$	$\frac{1}{2} \sum_X c_X z_X^2$
$K$	Equilibrium constant
$i$	Internal
$e$	External
$F$	In solid phase (absorbed by fibre)
$L$	In liquid phase (solution)

## EXPERIMENTAL

The experimental methods used were the same as in the previous paper<sup>1</sup>. Wool samples were

placed in acid mixtures for three days, and the absorption of hydrogen, chloride, and sulphate ions then determined from analysis of the mixtures. Table I gives the experimental equilibrium values of  $c$  and  $A$ . As there are no other ions present, we should find in all cases that

$$c_{\text{H}^+} - c_{\text{Cl}^-} - 2c_{\text{SO}_4^{2-}} = 0$$

In reality this quantity is generally not zero, but the difference is small, sometimes positive and sometimes negative, and thus probably a result only of experimental error. The absorption values obtained for pure acids are in good agreement with those of other workers<sup>1</sup>. Further, the calculated values

$$-\log f_{\text{H}^+} = \text{pH} + \log \theta_{\text{H}}$$

are in good agreement with the values from tables of  $f_{\text{H}^+}$  at the experimental ionic strengths. In calculating  $A$ , the "swelling" volume ( $v \approx 0.3$  ml./g. wool) was always regarded as free from solute (this assumption makes a negligible difference in the values obtained but renders the Donnan calculation a little simpler).

As in the previous paper<sup>1</sup>, we wish to compare these experimental results with those calculated from acceptable theories, viz. the Gilbert-Rideal, the Donnan, and also the ion-exchange theories.

## THE GILBERT-RIDEAL INTERPRETATION

The following three equations, deduced previously<sup>1</sup>, are employed—

$$\log \frac{\theta_{\text{H}}}{1 - \theta_{\text{H}}} + \log \frac{\theta_{\text{Cl}}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} - \frac{\log e}{RT} (\Delta \mu_{\text{H}} + \Delta \mu_{\text{Cl}}) = \text{pH} + \log a_{\text{Cl}} \quad (\text{i})$$

$$2 \log \frac{\theta_{\text{H}}}{1 - \theta_{\text{H}}} + \log \frac{\theta_{\text{SO}_4}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} - \frac{\log e}{RT} (2 \Delta \mu_{\text{H}} + \Delta \mu_{\text{SO}_4}) = 2 \text{pH} + \log a_{\text{SO}_4} \quad (\text{ii})$$

$$\theta_{\text{H}} = 2\theta_{\text{SO}_4} + \theta_{\text{Cl}} \quad (\text{iii})$$

pH values are taken from Table I, and values of  $f_{\text{Cl}}$  and  $f_{\text{SO}_4}$  from  $f$ -log  $I$  curves drawn from tabulated values<sup>1</sup>. Activities  $a = f c$  are then calculated for the chloride and sulphate ions from the  $c$ -values in Table I. From the experimental results of several authors<sup>1</sup> it has further been calculated that—

$$-\frac{\log e}{RT} (\Delta \mu_{\text{H}} + \Delta \mu_{\text{Cl}}) = 4.52 \quad (\text{iv})$$

$$-\frac{\log e}{RT} (2 \Delta \mu_{\text{H}} + \Delta \mu_{\text{SO}_4}) = 7.80 \quad (\text{v})$$

TABLE I  
REACTION WITH 0.1157 N-HCl + 0.1028 N-H<sub>2</sub>SO<sub>4</sub>

Sample	Dry Weight of Wool (g.)	Acid added (ml.)	Vol. of Soln. (ml.)	pH	$\mu_{\text{H}^+}$ ( $\times 10^{-3}$ )	$\mu_{\text{H}^+}$ ( $\times 10^{-3}$ )	$\mu_{\text{Cl}^-}$ ( $\times 10^{-3}$ )	$A_{\text{Cl}^-}$	$\mu_{\text{SO}_4^{2-}}$ ( $\times 10^{-3}$ )	$A_{\text{SO}_4^{2-}}$
I 1	1.0832	2	0	69.68	3.24	1.6	0.111	0.1	0.207	0
	0.9873	1	1	69.68	3.29	1.4	0.123	0.3	0.096	0.1
	0.9755	0	2	69.69	3.50	0.9	0.147	0	0	0.2
II 1	0.9712	4	0	69.67	2.79	3.7	0.211	0.8	0.419	0
	0.9746	2	2	69.68	2.98	2.6	0.263	1.6	0.123	0.1
	0.9794	0	4	69.68	3.23	1.6	0.306	0	0	0.6
III 1	0.8658	6	0	69.68	2.33	5.0	0.400	4.6	0.431	0
	0.9678	4	2	69.68	2.37	4.6	0.359	4.5	0.154	0.3
	0.9595	2	4	69.69	2.63	3.7	0.401	1.9	0.103	0.5
	1.0174	0	6	69.69	2.82	2.5	0.435	0	1.0	0.232
IV 1	0.9859	10	0	69.67	2.03	10.2	0.452	10.7	0.418	0
	1.0035	7	3	69.67	2.09	8.8	0.503	7.6	0.280	0.3
	0.9988	5	5	69.68	2.27	7.8	0.551	6.2	0.147	0.7
	1.0026	3	7	69.68	2.32	7.1	0.571	2.1	0.200	1.5
	1.0031	0	10	69.68	2.38	6.6	0.566	0	0	3.3
V 1	0.9876	20	0	69.67	1.67	24.3	0.629	25.7	0.531	0
	1.0374	15	5	69.67	1.74	22.5	0.658	21.4	0.236	1.4
	1.0495	10	10	69.67	1.84	21.5	0.655	15.4	0.080	3.8
	1.0055	5	15	69.67	1.90	20.6	0.684	7.8	0.036	6.9
	1.0071	0	20	69.67	1.97	19.6	0.685	0	0	10.2
VI 1	0.9586	35	0	69.66	1.39	48.5	0.701	45.6	0.912	0
	0.9864	28	7.05	69.71	1.45	46.6	0.726	38.9	0.535	3.0
	0.9774	20	15	69.66	1.49	45.2	0.723	28.1	0.305	7.6
	0.9565	15	20	69.66	1.52	44.6	0.716	21.5	0.249	11.0
	0.9843	7	28	69.66	1.59	43.3	0.683	8.0	0.257	16.2
	0.9644	0	35	69.66	1.66	42.0	0.697	0	0	21.2

The unknown quantities  $\theta_{\text{H}}$ ,  $\theta_{\text{Cl}}$ ,  $\theta_{\text{SO}_4^{2-}}$  in equations (i)–(iii) are then calculated by a method of successive approximation<sup>1</sup>. Finally the values of  $A_{\text{H}}$ ,  $A_{\text{Cl}}$ ,  $A_{\text{SO}_4^{2-}}$  are obtained from the relations  $A = A_{\text{max}}$ , where  $A_{\text{max}}$  is taken as 0.860.

A comparison between experimental and theoretical results gives very satisfactory agreement (Fig. 1 and 2).

In these calculations we have assumed that every sulphate ion is attached to only one positive site (Fig. 3a). However, we could make the alternative assumption that every sulphate ion is attached to two positive sites (Fig. 3b). To compare the calculated results in the two cases, we put  $t$  = total number of negative sites = total number of positive sites; and  $h$ ,  $r$ ,  $s$  = number of sites occupied by hydrogen, chloride, and sulphate ions respectively. In both cases (a and b) we have—

$$h_{\text{max}} = r_{\text{max}} = s_{\text{max}} = t$$

Further—

$$\begin{aligned} \frac{\theta_{\text{H}}}{1 - \theta_{\text{H}}} &= \frac{h}{t - h} \\ \frac{\theta_{\text{Cl}}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4^{2-}}} &= \frac{r}{t - r - s} \\ \frac{\theta_{\text{SO}_4^{2-}}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4^{2-}}} &= \frac{s}{t - r - s} \end{aligned}$$

Thus equations (i) and (ii) are the same in both cases, but the conditions for electrical neutrality are—

$$(a) \quad h = r + 2s$$

or, dividing through by  $t$ —

$$\begin{aligned} \frac{\theta_{\text{H}}}{h} &= \frac{\theta_{\text{Cl}}}{r} + \frac{\theta_{\text{SO}_4^{2-}}}{s} \quad (iii) \\ (b) \quad h &= r + s \end{aligned}$$

or

$$\theta_{\text{H}} = \theta_{\text{Cl}} + \theta_{\text{SO}_4^{2-}} \quad (vi)$$

Using equations (i), (ii), and (vi) instead of (i)–(iii), we are able to make a fresh calculation of the absorption of the three ions. However, this new assumption will also change the values of the affinity constants. For pure sulphuric acid, putting  $\theta_{\text{Cl}} = 0$  in (ii) and (vi)—

$$3 \log \frac{\theta_{\text{H}}}{1 - \theta_{\text{H}}} = - \frac{\log e}{RT} (2 \mu_{\text{H}^+} + \mu_{\text{SO}_4^{2-}}) - 2 \text{pH} + \log a_{\text{SO}_4^{2-}} \quad (vii)$$

The average value 8.32, from experiments by workers listed previously<sup>1</sup>, is substituted for 7.80 in (v), and used together with 4.52 in (iv). The calculated results in case (ii) are in reasonable agreement with the experimental results (Fig. 1 and 2), but not quite as good as the calculated values in case (a).

At low pH there should be some  $\text{HSO}_4^{2-}$  ions present. The binding of pure sulphuric acid has been calculated in the same manner as before<sup>1</sup>, taking this into account and assuming that every  $\text{SO}_4^{2-}$  ion is co-ordinated to two positive sites. But, as before<sup>1</sup>, we do not get better agreement with experimental values.

#### THE DONNAN INTERPRETATION

The method of calculation is as previously described<sup>1</sup>. However, as the swelling volume  $v$  is

here regarded as free from ions, the equations for the absorbed quantities (previously numbered (xix), (xxi), (xxii)<sup>1</sup>) become—

$$\frac{A_H}{e} = aH + [H^+]_0 + [HSO_4^-]$$

$$\frac{A_{Cl^-}}{e} = [Cl^-]_0$$

$$\frac{A_{SO_4^{2-}}}{e} = [SO_4^{2-}]_0 + [HSO_4^-]$$

and thus the previous equations (xxiii) and (xxiv)<sup>1</sup> are changed to—

$$\begin{aligned} 2f_{SO_4^{2-}} \frac{[SO_4^{2-}]_0}{e} \lambda^2 &= f_{SO_4^{2-}} \\ &+ \left( \frac{f_{Cl^-} [Cl^-]_0}{f_{SO_4^{2-}}} + \frac{2f_{HSO_4^-} [HSO_4^-]}{f_{SO_4^{2-}}} \right) \lambda \\ &= \frac{A_H}{e} = 0 \quad (\text{viii}) \end{aligned}$$

and if  $[HSO_4^-]$  is negligible—

$$\frac{2f_{SO_4^{2-}} [SO_4^{2-}]_0}{f_{SO_4^{2-}}} \lambda^2 + \frac{f_{Cl^-} [Cl^-]_0}{f_{SO_4^{2-}}} \lambda = \frac{A_H}{e} = 0 \quad (\text{ix})$$

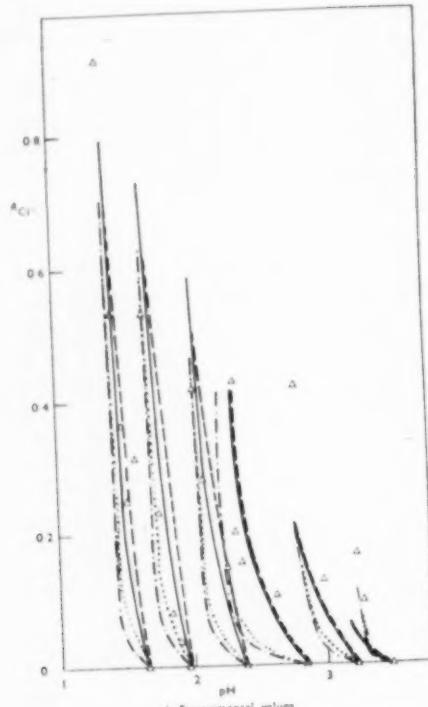


Fig. 1—Absorption of Chloride Ions

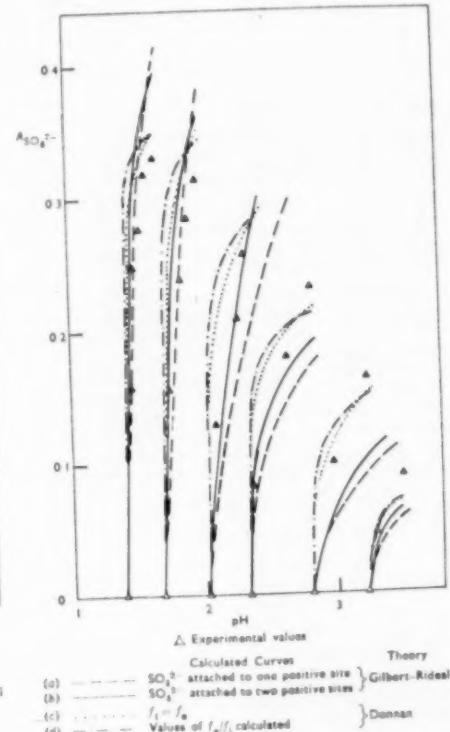
Values for  $e$  are taken from Peters and Speakman<sup>2</sup>. The experimental values for  $A_H$ ,  $[Cl^-]_0$ , and  $[SO_4^{2-}]_0$  (Table I) are used, and in the case of non-negligible  $HSO_4^-$  concentrations the quantities  $[SO_4^{2-}]$  and  $[HSO_4^-]$  are determined from the experimental  $e_{SO_4^{2-}}$  values and the dissociation constant for sulphuric acid. Values of  $\lambda$  are calculated according to (viii), assuming  $f_1 = f_0$  in all cases, and  $A$  values are calculated from (x) and (xi)—

$$\frac{A_{Cl^-}}{e} = [Cl^-]_0 + \frac{f_{Cl^-} [Cl^-]_0 \lambda^2}{f_{Cl^-}} \quad (\text{from previous equation (xvii)}) \quad (\text{x})$$

$$\begin{aligned} \frac{A_{SO_4^{2-}}}{e} &= [SO_4^{2-}]_0 + [HSO_4^-] \\ &= \frac{f_{SO_4^{2-}} [SO_4^{2-}]_0 \lambda^2}{f_{SO_4^{2-}}} + \frac{f_{HSO_4^-} [HSO_4^-]}{f_{HSO_4^-}} \lambda \quad (\text{xi}) \end{aligned}$$

For  $[HSO_4^-]$  negligible,  $f$  values are calculated from "internal concentrations"<sup>1</sup>, and are substituted in (xi); new  $\lambda$  values and hence  $A$  values are calculated from (ix)–(xi), and this series of successive approximations is repeated until there is no change in the  $f$  values (second approximation sufficient).

Fig. 1 and 2 give also the relation between these absorption values and the experimental values.



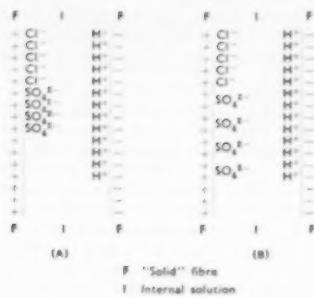


FIG. 3

The agreement is not so good as for Gilbert-Rideal plots, especially when calculated  $f$  values are used. The plots as far as  $[\text{HSO}_4^-]$  is considered are not given, nor the plots using  $A_H$  values from the Gilbert-Rideal theory. However, the deviations from the experimental plots are not significantly changed by these considerations.

#### THE IONIC-EXCHANGE INTERPRETATION

Another possibility is to consider wool at low pH values as an anion-exchange material. The reaction for chloride and sulphate ions may be written—



For equilibrium we have the general equation—

$$\frac{a_{\text{CL,L}}^2 \times a_{\text{SO}_4,\text{F}}}{a_{\text{CL,F}}^2 \times a_{\text{SO}_4,\text{L}}} = K \quad (\text{xii})$$

This equation may now be compared with the Gilbert-Rideal equation. Multiplying (i) by 2 and subtracting from (ii) we get—

$$\begin{aligned} \log \frac{\theta_{\text{SO}_4}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} - 2 \log \frac{\theta_{\text{Cl}}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} \\ = \log a_{\text{SO}_4} - 2 \log a_{\text{Cl}} - \frac{\log e}{RT} (4\mu_{\text{SO}_4} - 2\mu_{\text{Cl}}) \end{aligned} \quad (\text{xiii})$$

or

$$\begin{aligned} \log \left[ \frac{a_{\text{Cl}}^2}{a_{\text{Cl,L}}^2} \cdot \frac{\theta_{\text{SO}_4}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} \cdot \left( \frac{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}}{\theta_{\text{Cl}}} \right)^2 \right] \\ = - \frac{\log e}{RT} (4\mu_{\text{SO}_4} - 2\mu_{\text{Cl}}) \end{aligned} \quad (\text{xiii})$$

The correspondence between (xii) and (xiii) is clear, as—

$$a_{\text{Cl}} = a_{\text{Cl,L}} \quad a_{\text{SO}_4} = a_{\text{SO}_4,\text{L}}$$

$$\frac{\theta_{\text{Cl}}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} = a_{\text{Cl,F}}$$

$$\frac{\theta_{\text{SO}_4}}{1 - \theta_{\text{Cl}} - \theta_{\text{SO}_4}} = a_{\text{SO}_4,\text{F}}$$

It follows that the magnitude of  $K$  is given by—

$$\log K = - \frac{\log e}{RT} (4\mu_{\text{SO}_4} - 2\mu_{\text{Cl}}) \quad (\text{xiv})$$

From the values already given for (iv) and (v), we get—

$$(a) \log K = 7.80 - 2 \times 4.52 = -1.24$$

and

$$(b) \log K = 8.32 - 2 \times 4.52 = -0.72$$

for the sulphate ion attached to one and two positive sites respectively.

To compare the ion-exchange with the Donnan equation, (x) may be squared and then multiplied by (xi) ( $[\text{HSO}_4^-]$  being negligible), both equations being rearranged, to give—

$$\frac{f_{\text{Cl,L}}^2 [\text{Cl}^-]_0^2 \times f_{\text{SO}_4,\text{L}} [\text{SO}_4^{2-}]_0}{f_{\text{Cl,F}}^2 [\text{Cl}^-]_0^2 \times f_{\text{SO}_4,\text{F}} [\text{SO}_4^{2-}]_0} = 1 \quad (\text{xv})$$

Comparison with (xii) gives the relations—

$$f_{\text{Cl,L}} [\text{Cl}^-]_0 = a_{\text{CL,L}} \quad f_{\text{SO}_4,\text{L}} [\text{SO}_4^{2-}]_0 = a_{\text{SO}_4,\text{L}}$$

$$f_{\text{Cl,F}} [\text{Cl}^-]_0 = a_{\text{CL,F}} \quad f_{\text{SO}_4,\text{F}} [\text{SO}_4^{2-}]_0 = a_{\text{SO}_4,\text{F}}$$

and hence—

$$\log K = \log 1 = 0 \quad (\text{xvi})$$

The conclusion to be drawn here is that the ionic-exchange theory gives a general description of the equilibrium state, while both the Gilbert-Rideal and the Donnan theories give more detailed descriptions. There has been discussion of the definition of the  $a_F$  values in (xii), e.g. the values for the activities in the exchanger<sup>3</sup>. Here these values are defined in a different manner in the two special theories (cf. p. 510). But in both cases we can substitute experimental values in (xii) and calculate the corresponding values of  $K$ . By a statistical analysis we should then be able to compare these values with those predicted from the theories.

#### STATISTICAL INVESTIGATION OF THE EQUILIBRIUM CONSTANTS

The values of  $K$  are calculated from the experimental results in four different ways. Firstly, the Gilbert-Rideal symbolism is used according to (xii)

Sample	Calculated Values of $\bar{K}$			
	(a)	(b)	(c)	(d)
I 2	2.49	2.22	2.84	3.17
II 2	0.96	0.73	1.27	1.61
III 2	0.81	0.57	1.13	1.76
3	1.12	0.98	1.41	2.00
IV 2	0.79	0.64	0.98	1.67
3	0.52	0.45	0.77	1.46
4	2.08	2.22	2.23	2.95
V 2	0.26	0.13	0.52	1.28
3	-0.23	-0.28	0.11	0.89
4	-0.11	-0.09	0.20	0.99
VI 2	1.18	2.17	1.57	2.32
3	1.17	2.84	1.18	1.95
4	1.05	1.52	1.20	1.97
5	2.11	—	2.20	2.98
n	14	13	14	14
$\bar{x}$	1.01	1.08	1.26	1.03
$\sigma$	0.80	1.01	0.77	0.72
E	1.24	0.72	0	0
t	1.07	1.29	6.12	10.05

$n$  = Number of samples

$\bar{x}$  = Mean value

$\sigma$  = Standard deviation

E = Theoretical value

$$t = \frac{\bar{x} - E}{\sigma/\sqrt{n}}$$

and (xii) for the two cases of one sulphate ion attached to (a) one or (b) two positive sites (Table II), the values of  $A_{SO_4} \text{ max}$  being 0.860 and 0.430 respectively, and the other  $\lambda$  values being the same in both cases. Further, two calculations are made according to the Donnan theory, from (xii) and (xv), firstly taking  $f_{eff} = 1$  everywhere (c), and secondly using the calculated  $f$  values (d). We find that the distribution of the  $K$  values for a particular method of calculation is very asymmetric, but that the distribution of the values of  $\log K$  is fairly symmetrical\*. We therefore calculate mean values and standard deviations for  $pK$  ( $= -\log K$ ).

Application of the  $t$  test to compare these values with the theoretical shows that agreement is good in case (a) and reasonable in (b); in cases (c) and (d), however, the deviation is very significant (cf. Table III).

TABLE III  
Probability Limits for  $t$

(Degrees of freedom: $n = 14$ )		
Probability (%)	$n = 14$	$n = 13$
	$n - 1 = 13$	$n - 1 = 12$
90	1.77	1.78
95	2.16	2.18
98	2.65	2.68
99	3.01	3.06
99.9	4.22	4.32

Thus the Donnan theory is not applicable, and, if activity coefficients are considered, its failure is still more significant. But the Gilbert-Rideal theory is satisfactory, and the assumption of one site per sulphate ion is more probable than the assumption of two sites per sulphate ion. Further, we have noticed the small difference between the four (or the first three) values of  $x$ , in spite of the different methods of calculating "internal activity". If we compare the (a) and (b) values of  $x$  with the predicted Donnan value of zero, we get  $t$  values of 4.72 and 3.86, i.e. the Gilbert-Rideal method of calculating activities makes the experimental deviation from the Donnan theory smaller, although it is still very significant. On the other hand, the result of using Donnan concentrations can be compared with the predicted Gilbert-Rideal values. If  $x$  from (c) is compared with the  $E$  values 1.24 and 0.72 we get the corresponding  $t$  values 0.10 and 2.62, and if  $x$  from (d) is compared with the  $E$  values 1.24 and 0.72 we get  $t$  values of 3.59 and 6.30. In the latter case the deviation is very significant, i.e. calculated activity coefficients are not applicable. But the  $t$  value 0.10 gives very good agreement, i.e. we could use Donnan concentrations when applying the Gilbert-Rideal

\* It is obviously not quite normal, especially in case (b), but the deviations are not large, and an ordinary statistical analysis is justified.

theory. But this is not the case if the attachment of one sulphate ion to two sites is assumed, as  $t = 2.62$  corresponds to the 98% probability limit\*.

#### CONCLUSIONS

The essential conclusion from this investigation is that the Gilbert-Rideal theory should be used for calculating acid absorption. The reason for the failure of the Donnan theory is the assumption that the standard state of the chemical potential  $\mu$  for a certain ion is the same in the fibre as in the solution phase, i.e. in both internal and external phases. This assumption is well justified when the Donnan theory is applied to solutions separated by semi-permeable membranes. But in the present case the external and internal activities are not of the same character, and give a difference in standard chemical potential  $\Delta\mu$ , even if the external and internal concentrations are calculated in the same way (both calculated as Donnan concentrations). The cause of this difference in  $\mu$ , the specific ionic affinity, is not clear, but its existence makes the difference between dye anions and strong acid anions such as  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  a quantitative and not a qualitative one.

Another result of the difference in character between the "external" and the "internal" phase is that the ordinary concept of activity coefficient should not be applied to the fibre.

From the statistical analysis it may also be concluded that every  $\text{SO}_4^{2-}$  ion is probably attached to only one positive site, a conclusion that could not be drawn from steric considerations. This configuration is best seen by assuming the two negative charges of every ion to be placed symmetrically in relation to one positive fibre group, not placed symmetrically between two positive fibre groups. It is also possible that there is a statistical distribution between sulphate ions attached to one site and to two sites respectively, as the experimental equilibrium value lies between the theoretical values for these two cases.

\* \* \*

The author wishes to express his thanks to Professor Nils Gralén for interesting discussions and to Fru Birgit Olausson for her skilful laboratory work.

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(Received 26th May 1952)

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- \* Also, we must not use Donnan instead of Gilbert-Rideal concentrations when calculating single partition coefficients—  
 $R_{\text{Cl}} = a_{\text{Cl},\text{F}} a_{\text{Cl},\text{L}}$  and  $R_{\text{SO}_4} = a_{\text{SO}_4,\text{F}} a_{\text{SO}_4,\text{L}}$

## Some Aspects of the Drying and Heating of Textiles

### VII—The Stability of Thermal Modifications of Fibres in Subsequent Treatments

J. M. PRESTON and S. P. GUNDAVDA

Reduced swelling produced by the steaming of fibres has been investigated for its stability. Samples of steamed fibres have been given hot dilute acid and alkaline treatments. The reduced swelling of steamed viscose and cuprammonium fibres was found to be reasonably stable to the treatments given to them.

#### INTRODUCTION

In a previous paper<sup>1</sup> it was shown that suitable thermal treatments greatly reduce the swelling property of fibres. In the work described previously no attempt was made to establish the permanency of the changes produced. Both fundamentally and technically it is important to know whether the reduction of swelling is permanent to aqueous treatments or whether these cause the modified fibres to revert to their pristine condition.

#### EXPERIMENTAL

In order to test the permanency of the heat treatments, fibres were conditioned to the optimum

moisture content<sup>2</sup> and then heated at one of two temperatures for 1 hr. The temperatures selected were 100° C. and 135° C. Viscose, cuprammonium, and silk fibres were tested.

The treated and untreated samples were tested for their water retention-swelling values using the normal centrifuge technique<sup>3</sup>. Subsequently, treated samples were boiled for 1 hr. in distilled water, in dilute hydrochloric acid of pH 1.8-4, or in dilute alkali of pH 10-12. After alkaline treatments samples were given a wash in cold dilute acid and a wash in water. Subsequently the samples were again tested for their water retention-swelling power without further drying.

The results shown in the Table were obtained.

#### DISCUSSION

Boiling the regenerated cellulose rayons in distilled water produced small or negligible changes in the water retention-swelling values. The most marked change occurred in the case of the untreated cuprammonium fibres, where there was a significant reduction of swelling. In the case of the treated viscose fibres, boiling at pH 1.8 produced a significant reduction of swelling. Boiling in alkaline solutions produced varying results. The fibres treated at 100° C. had their swelling reduced, whilst those treated at 135° C. showed a small increase of swelling.

Smaller changes of swelling were produced by boiling the cuprammonium fibres than by boiling the viscose fibres in the different solutions. On boiling in distilled water or alkaline solution silk fibres show a marked tendency for the swelling values to revert to those of the untreated fibres. Boiling in acid solution produces smaller changes.

The general conclusions to be drawn from these experiments are that the changes of water retention-swelling produced by thermal treatments are reasonably stable in the case of the regenerated cellulose fibres. In this they differ from the silk fibres.

\* \* \*

Since this work was done, one of us (S. P. G.) has found some indication of increased hydrogen bonding in cellulose fibres after heating, which should be investigated further.

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THE UNIVERSITY  
MANCHESTER

(Received 5th December 1951)

#### References

FIBRE	Treatment	Water Retention	
		Heated at	Then boiled in
	(°C.)	Liquid of pH	(%)
VISCOSE RAYON	—	—	105
	—	(7)	103
100	—	—	95
	(7)	—	93
	1.8	—	85
	10	—	91
	12.4	—	92
135	—	—	80
	(7)	—	81
	1.8	—	76
	10	—	81
	11	—	82
	12.8	—	83
CUPRAMMONIUM RAYON	—	—	90
	—	(7)	86
100	—	—	69
	(7)	—	70
	1.8	—	69
	10	—	67
	12	—	67
135	—	—	55
	(7)	—	55
	1.8	—	56
	10	—	57
	12	—	57
SILK	—	—	54
	—	(7)	55
100	—	—	45
	(7)	—	50
	1.8	—	47
	9.8	—	53
135	—	—	37
	(7)	—	52
	1.8	—	45
	9.8	—	53

<sup>1</sup> Preston, Nimkar, and Gundavda, *J.S.D.C.*, **67**, 169 (1951).

<sup>2</sup> Preston, Nimkar, and Gundavda, *J. Textile Inst.*, **42**, p. 79 (1951).

## CORRESPONDENCE

*The Editor does not hold himself responsible for opinions expressed by correspondents.*

### The Effect of Copper in Wool Dyeing

It is well known that copper can react with many chrome dyes to form a copper lake which is frequently of a different colour from the desired chrome lake, and that sufficient copper to affect the shade may be derived from copper-containing parts of the machinery. However, it may not be so well appreciated how much effect traces of copper already present on the wool may have on dyeing. In view of the increasing use of copper sulphate in sheep dipping, experiments have been made to find how much copper may be expected to be retained by the wool and how great an effect it would have on dyeing.

It has been found that weak, e.g. 0.15%<sub>w/w</sub>, dyeings of some chrome dyes are perceptibly affected by traces of copper of the order of 15 p.p.m., which might normally be expected in wool, while 70 p.p.m. copper (derived from copper-contaminated carbonising acid) causes a very marked change of shade in a 0.5%<sub>w/w</sub> dyeing of Solochrome Brown M (metachrome).

It is suggested that the well known difficulty of reproducing pastel shades dyed with chrome dyes such as the Solochrome Brown M and Solway Blue Black B types may be partly due to the presence of traces of copper, varying in amount from batch to batch and so affecting the dye to different extents. If this is the case, the use of a sequestering agent such as ethylenediaminetetra-acetic acid as a routine measure should prove effective in improving the batch-to-batch uniformity and reducing the time taken for dyeing.

As copper sulphate is now being widely used at a low concentration (0.03%<sub>w/w</sub>) as a bacteriostat in Gammexane sheep-dips and to a limited extent at a higher concentration (0.2%<sub>w/w</sub>) for dipping against a skin disease called mycotic dermatitis, besides being very widely used at a very high concentration in footrot baths, which sometimes accidentally contaminate the wool, the normal copper content of wool may be expected to increase a little, while occasional batches of wool may have a quite appreciable copper content. Should one of these batches be dyed in a light shade with one of a number of chrome dyes, an unexpected result would be obtained.

L. F. STORY

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NEW ZEALAND

3rd October 1952

### Production of Compendia

The paper by Dyson<sup>1</sup> in which easily derived ciphers are described, by means of which organic compounds may be indexed without reference to trivial names, leads us to wonder whether this may not be the answer to the vexed question of Beilstein, the *Colour Index*, and similar monumental compilations. Reference works such as

these are expensive to produce and to buy (especially Beilstein), and the very magnitude of the task of revision and incorporation of newly accumulated information makes new editions very infrequent and inevitably out of date.

These works could be published as systems of filing cards, arranged in cipher order, with one card for each compound (printed on both sides, or running to two cards in some cases). An overall alphabetical index, which might also take the form of small cards, would serve to connect the trivial names with the ciphers.

The advantages of such a revolutionary publication would be many. A card index system kept by the publishers would allow the continual and systematic accumulation of new information in a form which would make it comparatively simple to issue new batches of cards yearly, or half-yearly, thus avoiding the high cost and time-lag associated with the revision of a whole book, and the buyer would simply insert the new cards in his files. When sufficient new information became available to warrant it, new cards could be prepared and published for existing compounds, to replace the old ones.

Though the initial cost of cards and filing cabinets might be a little high, this would amply repay itself over the years, as the system would be permanent, the access to information rapid and systematic, and the cost of the new cards necessary to keep the files abreast of developments comparatively small. No conventionally printed book could afford such advantages, even if kept reasonably up to date by supplements.

No doubt means could be found to reduce the cost of such a production: for instance, the publishers might cross-index the whole field in terms of spheres of interest, and thus offer in addition to a basic selection (comprising the bulk of the compounds) separate collections of special-interest to workers in particular fields such as plastics production, biochemistry, dye manufacture, and foodstuff processing. Data concerning closely related substances such as the alkyl esters of an acid might be printed on one card, the alphabetical index giving cross-references to the single card in the cipher file.

We feel that some system such as this, providing as it does for infinite and systematic expansion, would provide a solution to the present highly unsatisfactory position.

J. W. DUARTE

A. E. STUBBS

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<sup>1</sup> Dyson, G. M., *Chem. and Ind.*, 676 (12 July 1952); cf. p. 544 of this issue;  
Idem, *ibid.*, 1081 (1 Nov. 1952).

## ERRATUM

**Continuous Stripping of Carpet-yarn Waste.** P. J. Wood and E. T. Duffy (*I.S.D.C.*, 68, 38 (Jan. 1952)).—The phrase "56% of 3% acetic acid" should read 3% of 56% acetic acid.

## Notes

## Proceedings of the Council

At a meeting of the Council held at the offices of the Society, 19 Piccadilly, Bradford, on 8th October 1952, the proceedings included the following items of interest—

**ANNUAL CONGRESS OF THE A.C.I.T.**—Dr. T. Vickerstaff reported that he, as well as Dr. D. P. Raper and Mr. E. A. Swift, had attended the Annual Congress of the Association des Chimistes de l'Industrie Textile, held in Toulouse on 26-28th September 1952. The congress had been very well organised, and both scientific and social sides had proved very successful.

**THIRD GEORGE DOUGLAS LECTURE**—It was reported that Dr. T. Holbro had accepted an invitation to deliver this lecture, giving a survey of the modern approach to dyes which would cover up-to-date applications and uses as well as the search for new dyes.

**S.D.C. OF AUSTRALIA**—It was reported that a letter had been received from Mr. W. C. Castle, President of the Society of Dyers and Colourists of Australia, conveying greetings to our President and Council. The inaugural meeting of a Queensland Section was to be held in Brisbane on 25th August 1952, making four sections in all. The greetings had been warmly reciprocated, and our good wishes offered for the success of the new venture.

**HISTORICAL RECORDS COMMITTEE**—It was resolved to invite Mr. R. Brightman to serve on this committee.

**MEMBERSHIP**—Fourteen applications for ordinary membership and two for junior membership were approved.

Meetings of Council and Committees  
November

Council—12th

Publications—18th

*Colour Index* Editorial Panel—19th

Terms and Definitions—19th

Diploma—12th

Examinations Subcommittee—26th

## Death

We regret to report the loss by death of Mr. F. Hern.

## Third George Douglas Lecture

The lecturer will be Dr. T. Holbro, of Basle, Switzerland, and he will speak on *The Search for New Dyes in relation to Modern Developments in the Textile Field*. Arrangements have been made for the lecture to be delivered in the Chemistry Lecture Theatre, University of Leeds, late in March 1953.

## Tenth Mercer Lecture

The subject of the Tenth Mercer Lecture will be *The Coloration of Leather*, and the lecturers J. R. Blockley, M.Sc., F.R.I.C., and D. H. Tuck, A.L.C., both of the National Leathersellers College. The lecture will be held on Thursday, 9th April 1953 (the eve of the Society's A.G.M. and Dinner), at 7.30 p.m. in the Royal Institution, Albemarle Street, London W.1.

## Summer School 1952

The third Summer School of the Society was held at Loughborough College, Leicestershire, during the week 23rd-30th August 1952. The subject was *Background to the Industry*, and 60 students were enrolled, 54 of them resident in the Dower House and Soar House hostels of the College at Quorn. Included among the resident students were visitors from Norway, Holland, Australia, Italy, and Belgium. The School opened with a social "get-together" on the Saturday evening. A tour of the Charnwood Forest area followed on the Sunday.

During the week following, ten lectures were given during the morning periods, while in the afternoons the students split into groups which attended nine laboratory demonstrations and nine works visits. The School was closed on the following Saturday by the President (H. H. Bowen, Esq.), following a valedictory address by F. L. Goodall, Esq.

The students generally have expressed their satisfaction with the course, and the Committee would like to extend its sincere thanks to the lecturers, demonstrators, and Loughborough College staff, who worthily maintained the high level of previous Schools; to the Midlands industrialists who threw open their works; and to the very many voluntary workers who helped to make the week memorable both for its pleasant atmosphere and for its efficiency.

The Committee responsible for the organisation consisted of Messrs. W. A. Edwards (Chairman), C. W. Edwards (*Honorary Secretary*), A. W. Carpenter, A. Datyner, G. S. Egerton, R. L. Elliott, A. Hout, A. P. Kershaw, H. C. Olpin, C. B. Stevens, and A. G. Tyler.

Council has recorded its gratitude and congratulations to all associated with the organisation of the Summer School, and especially to local members of the Society.

## Worshipful Company of Dyers

Captain Cyril B. Tidd, R.N., has been elected Prime Warden, and Mr. Cyril R. Egerton Renter Warden.

### Worshipful Company of Feltmakers

The new officers elected in October are—

G. C. Bourne, J.P.	<i>Master</i>
J. Begley	<i>Senior Warden</i>
Colonel Sir G. Christie-Miller, K.C.B.	
D.S.O., M.C., D.L.	<i>Renter Warden</i>
Major F. J. Johnson	<i>Third Warden</i>
E. R. Laycock, O.B.E.	<i>Fourth Warden</i>

Arrangements have been made to provide all the officers with the type of hat depicted in the Company's coat of arms, for wear on ceremonial occasions.

### Society of Dyers and Colourists of Australia

#### Fourth Annual Report (1951-52)

This has again been a successful year, and the year's activities are summarised—

**COUNCIL**—The Council met twice, and at the first meeting unanimously re-elected Mr. John A. Eastwood as President for a second term of office. The Council approved publication of amended Bye-laws, and these are now available to each member.

**MEMBERSHIP**—This continues to rise slowly, and a number of new members from Queensland were admitted during the year.

**MONTHLY MEETINGS**—Sectional committees again provided attractive programmes, and this reflects great credit on those gentlemen concerned. Our thanks are extended to all who gave up their time to prepare and deliver lectures—we are indeed grateful.

**SOCIAL**—The Social Committees are to be congratulated for their efforts in organising the various functions that have become a permanent part of our activities—Annual Ball, Dinner, and Golf Day. The Victorian Section, being more fortunately placed than the other two sections, again visited a county centre for a weekend, this time Wangaratta. An inspection was made of the factory of Bruck Mills Ltd., and we ask the management to accept our thanks. Other activities were also organised, and a most enjoyable week-end was had by all attending.

The President's Golf Trophy again resides with the New South Wales Section, who were indebted to Mr. A. C. Sandiford for retaining the trophy. The winner was decided on a count back, a very close contest.

**LIBRARIES**—The Honorary Librarians have continued their diligent work throughout the year, and a number of new books have been added.

**SECTIONAL COMMITTEES**—They have been most active again, as is very evident from earlier remarks. The Council thanks all members who have served on these Committees.

**PUBLICATION**—Through the Publication Sub-committee the Council has endeavoured to keep this important side of our activities going, and has performed very creditably. When there was a shortage of local material, we were indebted to the Society of Dyers and Colourists for permission to reprint articles from their *Journal*. The Council wishes to place on record its thanks to the authors of various papers who permitted them to be published, and also to a number of other contributors.

**SUMMARY**—In conclusion I would say that the future of the Society is assured, and we can look forward to a most successful 1952-53. The President—a Life Member of the Society—has been an untiring worker, and visited all sections during his term of office. To all other members of the Council and Secretaries of sections, I thank you for your co-operation during my term of office.

D. A. LANGSHAW  
*Honorary General Secretary*

The office of Honorary General Secretary of the Australian Society is now occupied by Mr. B. Firth, of 5 Fordholm Road, Hawthorn E.2, Victoria, Australia.

### Manchester College of Technology

The Jubilee of the College was celebrated in October 1952, although it is a direct descendant of the Mechanics Institution founded in 1827. The total of part-time and full-time students reached a maximum of 8558 in 1949, and the number of full-time university students rose to 880 in 1950. Extensions to the present buildings, including a new dyehouse, are estimated to cost £1,680,000, and will nearly double the accommodation now available.

### Opening of New W.I.R.A. Buildings

Three new laboratory blocks were formally opened on Friday, 17th October 1952, by Viscount Swinton, P.C., C.B.E., C.H., M.C., Minister of Materials, at "Torridon", Headingley, Leeds 6, the headquarters of the Wool Industries Research Association. Displays in the various laboratories explained the work of the Association. The departments which staged displays illustrating their work included physics, testing machines, quality control and statistics, electronics, moth control, drying, dyeing and finishing laboratories, and dyeing and wet-finishing plant. An exhibition was arranged in co-operation with the International Wool Secretariat, showing the processing of wool.

### Nobel Prize for Chemistry

The Nobel prize for chemistry for 1952 has been awarded to Dr. A. J. P. Martin, F.R.S., and Dr. R. L. M. Syngle, F.R.S., biochemists at the National Institute for Medical Research, London, and the Rowett Research Institute, Aberdeenshire, respectively, who worked together on partition chromatography at the Wool Industries Research Association from 1940 to 1946. Dr. Martin contributed a paper to the Society's symposium on *Fibrous Proteins* held in Leeds in 1946.

### Exhibition of Textile Printing

An exhibition showing the history of textile printing will be held in the Rijksmuseum Twenthe, Enschede, from the middle of January to the middle of February 1953, and will include medieval and oriental prints, old recipe books, old hand-blocks, etc. Further information can be obtained from the Enschedese Kunststichting, J. P. Sweelinckstraat 34, Enschede, Holland.

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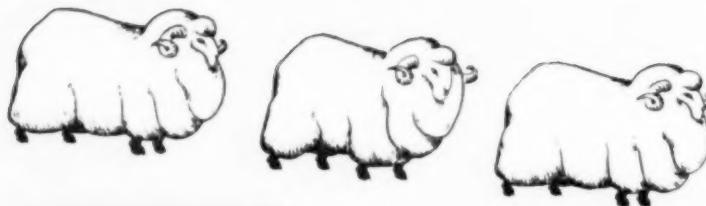
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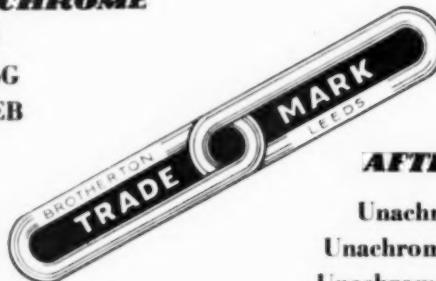


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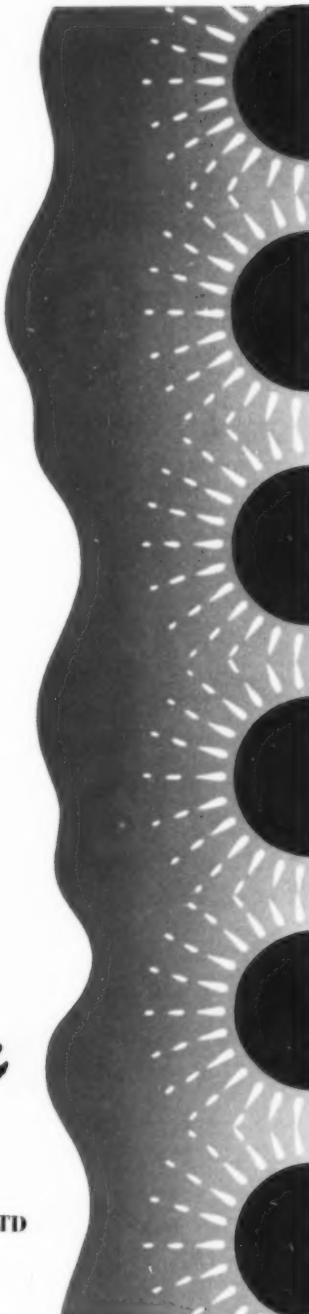


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and  
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LEEDS



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unrivalled in brilliancy and fastness properties



FARBENFABRIKEN BAYER LEVERKUSEN



### Emil and Otto Fischer

These cousins, born on 9th October and 28th November 1852 respectively, collaborated in Munich in establishing (1878) the Rosaniline dyes as derivatives of triphenylmethane. The more famous Emil Hermann had obtained his doctorate, after working under A. von Baeyer at Strasbourg, for a thesis on fluorescein and orcinolphthalein. The following year (1875) he first prepared phenylhydrazine, by reduction of diazonium solutions, and subsequently employed this reagent in his well

known work on the sugars. Another great contribution of Emil to organic chemistry lay in the field of polypeptides, interest in which is very active at the present time. Otto Philip was the first to synthesise Malachite Green, and his name is commemorated in the Fischer-Hepp rearrangement of *N*-nitrosoamines to the corresponding nucleophilic nitrosated bases. An obituary notice of Otto Fischer was published in the *Journal* in 1932 (48, 233).

### OBITUARY NOTICE

#### Oswald Gunnell

By the death of Oswald Gunnell the dry cleaning industry has lost one of its outstanding personalities. The fifty-six years which he spent at Johnson Brothers saw that firm grow from a very modest size to one of the largest dyeing and dry cleaning concerns in the world. During that expansion, his wide scientific knowledge and keen judgment were invaluable.

After being educated at the Yorkshire College, he went to Johnson Brothers as works chemist, but his abilities as a sound organiser were soon recognised, and he was transferred to management, finally becoming personal assistant to Sir Benjamin Johnson. On the death of Sir Ben, Gunnell was appointed Chairman of Johnson Brothers and also Chairman of the Johnson Group of Companies.

He was by nature shy and retiring, and outwardly appeared reserved and difficult to approach.

On closer acquaintance, however, one soon realised his charm and friendliness, and to all the employees at Johnsons' he was not a remote figure but a well loved and admired colleague. Each Christmas he gave a party for all the children of his employees, and here he was seen at his best.

He served for many years on the Council of the National Federation of Dry Cleaners, and was its president on several occasions. Although he took a keen interest in the Society, geography prevented his playing a very active part in its affairs. When his colleague, the late F. F. Flinn, was honoured by the Presidency of the Society, Mr. Gunnell was delighted and gave Mr. Flinn his most active support.

We, of the Johnson Group, will miss the wise guidance of Mr. Gunnell, and mourn the passing of a friend and colleague.

A. BREARE

### New Books and Publications

#### The Chemical Revolution A Contribution to Social Technology

By A. Clow and Nan L. Clow. Pp. xvi + 680 + frontispiece. London: Batchworth Press. 1952. Price, 50s. 6d.

This most important as well as most interesting and most readable book is unlike any other work so far published. By surveying the development of agriculture and industry in Scotland during 1750-1830, the authors show that the Industrial Revolution which occurred in Britain during that period was as much chemical as mechanical; for example, without the introduction of bleaching with chlorine and advances in metallurgy, the inventions of mechanical spinning and weaving could not have developed at either the speed or on the gigantic scale that they did. It is also true that they owed much to the immense supplies of cotton grown by slave labour in North America. The vital rôle that chemists played in the development of the cotton industry in Scotland is amply illustrated in such chapters as VIII—Cognates to the Textile Industry, IX—The Scottish Bleaching Industry, X—The Philosophy of Colour, XI—Calico-printing, and XII—Mordants and the Macintoshes. The authors spent much time in

consulting original literature, and the references alone make it invaluable to historians of technology.

The authors' work has been done excellently, and they cannot be too highly complimented on it; in manuscript it won the Senior Hume Brown Prize of the University of Edinburgh, and there is no doubt but that it will become a classic. Every member of this Society will find the book of absorbing interest, but it can be warmly recommended to a much larger public. It ought above all to be read by writers of school history books, for every child is taught about Watt, Crompton, Arkwright, and Cartwright, but few, if any, are ever told about Scheele, Berthollet, Leblanc, Macintosh, or Tennant; yet without these chemists the work of the engineers would have been in vain, and the cotton industry owes just as much to the latter group as to the former. The book is well produced and remarkably free from errors. In future printings and editions the diagram on p.175 should have the reference to Animal Fibres (Wool) deleted, for not one of the agents mentioned—lime, bleach liquor, and bleaching powder—was or is used in bleaching wool. The price may seem high, but it is less than 1d. per page, and the book is worth every penny of it.

C. O. CLARK

## Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.  
Any publication abstracted may be referred to by members of the Society on application to  
Dr. C. B. Stevens, Dyeing Department, Leeds University

### Badische Anilin- & Soda-Fabrik

**PALATINE FAST DYES ON WOOL CLOTH.** This pattern card contains thirty-five single dyes in two depths and fourteen mixtures together with the relevant methods of application and fastness data.

### PERLITON AND PERLITAZOL DYES ON PERLON (NYLON)

Twenty-nine dyes in two depths are shown in this card. These ranges of disperse dyes have been developed specifically for dyeing Perlon and nylon in the form of hosiery and drapery where level-dyeing properties and good fastness to light are the main requirements. If maximum fastness to washing is required, this class of dye, with the exception of Perlitzol STN, should not be used.

**BAASF TEXTILE AUXILIARIES.**—This book is a guide to the use of the complete range of textile auxiliary products produced by the Baasf. The contents are divided into two main sections: firstly, a survey of the products available for use in the manufacturing, dyeing, and finishing of (a) wool and mixtures of wool with other fibres and (b) cotton, rayon, linen, and mixtures thereof; secondly, an alphabetical list of the products with the more important data and brief notes concerning their nature, properties, and uses. The 250 pages of text contain a large number of illustrations, including photographs, line drawings, and diagrams.

**INDANTHREN, ANTHRA AND BRILLIANT INDIGO DYE-STUFFS.** SCREEN PRINTS BY THE COLLORESENE PROCESS.—This is a particularly elaborate pattern card, showing a range of fifty-four vat dyes printed in two depths on cotton spun rayon mixture fabric, and containing almost nine large patterns including cotton, cotton cretonne, honeycomb rayon, and rayon repp printed with mixtures of dyes on vat-dyed grounds. The Colloresene printing process is essentially a pigment technique in which the dye is printed together with a special thickener which will coagulate in alkali. After printing and drying, the fabric is padded with an alkaline solution of the reducing agent, steamed, and finished. The text includes full working details together with data on fastness to light, washing, and chlorine on cotton and cellulose rayon and suggested uses for each dye.

**INDANTHREN "COLLOSONOL" DYE-STUFFS.**—This leaflet lists the first twelve members of the Collosonol range of vat dyes. They are characterised by extremely small and uniform particle size, being considerably finer than the Powder Fine Type 8039 brands hitherto recommended for pigment padding. Aqueous dispersions of the new products are similar in behaviour to colloidal suspensions. Detailed information on application and properties is included.

**INDANTHREN GOLD ORANGE G SUPRAFAX PASTE.**—This new vat dye is intended to replace the Printing Orange GO brand, to which it is superior in ease and rapidity of fixation and resistance to frost. It is printed according to the normal potash-Rongalite process, the addition of Glycine A being recommended to increase colour yield. In hue and fastness properties it is identical with the product it replaces. Fastness figures on cotton include—Light 7, washing at 100° C. 4-5, chlorine 5.

**PALATINE FAST YELLOW 6 GEN.**—This premastilled dye gives greenish yellow, and is particularly recommended for use alone and in mixtures on all types of wool materials. Its dyeing characteristics and fastness properties are of the same order as those of the other members of the range. Fastness figures include—Light 6, washing 4, perspiration 3-4.

**RONGALITE CL.**—A discharging agent for use in printing, especially on vat-dyed grounds.

**SOL DEVELOPER GAN.**—A dye solvent for use in textile printing.

**HYDROSULPHITE CONC. BAASF.**—A reducing and stripping agent for use in the textile industry.

### Cassella Farbwerke Mainkur

**IMMEDIATE AND IMMEDIATE LEUCO DYE-STUFFS ON SUÈDE LEATHER.**—This pattern card (German text) shows a

range of twelve Immediate and eleven Immideal Leuco dyes on leather, together with nine two- and three-component mixture dyes produced with dyes from both ranges.

### Clayton Aniline Co. Ltd.

**DYEING PROPERTIES OF ACID AND CLOTH FAST DYES.**—This circular contains the tabulated results obtained when the dyeing properties of the complete ranges of acid and Cloth Fast dyes were examined on wool using the testing methods prescribed in the Report of the Society's Wool Dyes Committee, whose detailed recommendations for testing are given in the text.

**FASTNESS PROPERTIES OF ACID, CLOTH FAST, AND DIRECT DYES ON WOOL.**—This circular contains the tabulated results obtained when the fastness properties on wool of the complete range of acid dyes were assessed according to the testing methods recommended by the Society's Fastness Tests Committee. Full details of each method of testing are given in the text.

**FASTNESS PROPERTIES OF CHROME DYES ON WOOL.**—This circular is identical in scope and intention with the preceding one, and covers the complete range of chrome dyes dyed by each method where applicable.

**EFFECT OF NON-SHRINK PROCESSORS ON ACID, CLOTH FAST, AND DIRECT DYES ON WOOL.**—This circular contains the results obtained when dyeings of the complete ranges of the above dyes were subjected to the Dylanz, Epilox, Negafel, papain, and Woolindras shrink-resist processes.

**CHACET DYE ON ACETATE RAYON MATERIAL.**—This card contains dyeings in two depths of sixteen disperse dyes suitable for use on acetate rayon and mixtures containing this fibre.

**CHLORASTINE FAST BROWN 8 RLL.**—This direct dye gives red browns, of very good fastness to light, which withstand crease-resist finishing, and is of particular interest alone and in mixtures for dyeing furnishing fabrics. It is a class C dye.

**CHROME FAST BORDEAUX FRL.**—**SYNCHROMATE BORDEAUX FBL.**—This chrome dye is recommended for application by single-bath, afterchrome, or on-chrome methods to give dyeings somewhat bluer than those obtainable with Chrome Fast Bordeaux B. It is suitable for dyeing loose wool, yarn, and cloth in open vats and circulating liquor machines. It is also of interest for direct printing on wool and silk including the printing of slubbing. Fastness figures on wool using the Synchromate method include—Light 6-7, milling (soap and soda) 4-5, washing (at 50° C. using soap and soda) 4-5.

**CHANONE BLACK DRB.**—This vat dye gives dark greys to full blacks of very high fastness to light, soda boiling, and peroxide on cellulose materials by direct dyeing. It is easily reduced to a readily soluble leuco compound, and is thus suitable for dyeing in circulating liquor machines. Greys are more advantageously produced by pigment padding. Dyeings are unaffected by crease-resist finishing. Fastness figures on cotton include—Light 7-8, chlorine 4-5, soda boiling 5.

**CHANONE BRILLIANT PINK G.**—Pinks and reds of excellent fastness to light and piece bleaching are given by this vat dye, which may be applied on the jig, in circulating liquor machines, and by pigment padding. It is particularly recommended for furnishing fabrics and goods which are intended for subsequent rubberproofing.

**CLOTH FAST BRILLIANT RED 3 BW.**—This acid dye gives bright bluish reds of good fastness to wet processing. It is applied from weakly acid or neutral baths to give dyeings which are fast to carbonising, unaffected by chrome in the dyebath, and unaffected in hue by prolonged boiling. It is also of interest on silk and nylon. Cellulose and acetate rayon effects are reserved white. It is suitable for direct printing and for ground shades for coloured discharges on wool and silk. Fastness figures on wool include—Light 4-5, washing (at 50° C. with soap and soda) 4-5, heavy milling 4-5.

**NEOLAN BLACK WA EXTRA**—This premetallised dye is recommended for blacks of good wet fastness on all forms of wool materials. It is also of interest for printing wool, silk, and cellulosic rayons, but it is not dischargeable. It is of value for dyeing leather, particularly as a basis for greys. Fastness figures on wool include—Light 7, milling 4-5, washing 5.

**PYROGENE PURE BLUE BL**—This sulphur dye gives dyeings of very good fastness to light for blues of this type. It is readily soluble and is principally of interest for dyeing cotton and cellulosic rayons. Aftertreatment of dyeings with chrome and copper sulphate makes them greener and duller without significantly affecting the fastness. Fastness figures on cotton include—Light 6, washing 2, perspiration 2-3.

#### Durand & Huguenin S.A.

**INDIGOSOL GOLDEN YELLOW AR**—This is a solubilised vat dye giving on cellulosic materials golden yellows which are completely dischargeable. It has only moderate substantivity in the absence of electrolytes, and is thus very suitable for application by padding; but it can be dyed on the jigger, in the beak, or in circulating-liquor machines if Glauber's salt is added. It is readily developed, and is not sensitive to overoxidation. Fastness figures on cotton include—Light 6, chlorine 5, soda boiling 3-4.

**INDIGOSOL ORANGE ISR**—This solubilised vat dye is recommended for fast printing styles on cellulosic materials, especially for padded ground shades on goods printed with vat dyes, since it is easily reserved with reducing agents such as sodium hydrosulphite or thiosulphate, and the use of agents such as Reserve X is unnecessary. Dyeings are dischargeable only to a poor wash. Fastness figures on cotton include—Light 7, washing (1 hr. at 100°C.) 4, soda boiling 4-5.

#### Farbenfabriken Bayer

**INDANTHREN RED F3B**—This vat dye gives bluish reds and is particularly recommended for use on furnishing fabrics and yarns to be subsequently bleached in the piece. The Powder Superfine brand is suitable for padding, and a Type No. 8015 is available which is sufficiently free from copper and manganese for use on materials to be rubberproofed. It is suitable for pale dyeings on silk and also for printing by the Rongalite, potash and Colloresin processes. Fastness figures on cotton include—Light 7, chlorine 5, soda boiling 4-5.

#### Imperial Chemical Industries Ltd.

**S.C.C. 15 OLIVE DRAB ON COTTON CANVAS**—This pamphlet describes the production of the above shade using vat dyes and either dyeing on a mineral khaki base or overdyeing with mineral khaki after vat dyeing. The Carbindone Black B, which is a component of the recommended mixture, is vatted separately at the boil with sodium sulphide.

**CHLORAZOL COPPER BLUE 2B**—This direct dye is recommended for blues on cellulosic fibres. Aftercoppering with copper sulphate readies the colour and considerably improves the fastness to light of the dyeing. Crease-resist finishing slightly reduces the fastness to light, the colour becoming slightly greener and weaker. Although of very limited interest for direct printing, it is suitable for both white and coloured discharges on cotton and for coloured discharge work on viscose rayon. Fastness figures on cotton of a dyeing of standard depth aftertreated with copper sulphate include—Light 5, washing (repeated mild, five times at 40°C.) 2-3, water 4.

**SUGGESTIONS FOR MINIMISING "MOTTLING" WHEN USING DIRECT COTTON DYES FOR PAPER COLORATION**—This technical circular discusses the incidence of "mottling" when using different methods of colouring paper, and on the basis of extended laboratory and bulk trials suggests that the best method of avoiding mottling is to add the dye as early as possible in the heating cycle, with as long an interval of time as possible between the dye addition and the final sizing. Addition of rosin soap may be an advantage, but it is most important that addition of the alum be delayed as long as possible when using dyes liable to give mottled results. To facilitate the selection of single dyes and mixtures, a range of forty-nine direct dyes have been examined and graded on a 1-5 scale according to their tendency to mottle.

**WOODSTAINS**—This pattern card describes the colouring of wood using water-soluble dyes, aqueous dispersions of pigments, spirit/alcohol soluble dyes, dyes soluble in solvent naphtha, and dispersions of selected pigments in this solvent; this last is a laboratory development, and the details given are recommendations only. Each type of stain is shown on white sycamore veneer, one half of the pattern in each case being unfinished and the other treated with a spirit shellac lacquer. For each type of stain, selected dyes are illustrated together with examples of typical mixtures, and the scope and limitations of each technique are detailed in the text.

**NOLEDON GREY B**—This is a solubilised vat dye recommended for producing bluish greys on cellulosic materials, particularly where high fastness is required together with good penetration. It is developed by the normal acid oxidation technique, and may be applied in circulating-liquor machines, on the jig, or by padding methods. It is of interest on silk and may be applied to wool, but it is only of limited use on this fibre. Fastness grades on cotton include—Light 5, washing (repeated severe, five times at 100°C.) 3-4, hypochlorite 3-4.

**CALEDON PRINTING YELLOW 6 GS PASTE**—This vat dye gives bright greenish yellows of good wet fastness and higher fastness to light than existing yellows of this hue when applied to cellulosic materials by any of the normal printing methods. It is particularly suitable for use in conjunction with Caledon Printing Jade Green XN. Ground shades are dischargeable to a white by the Formosol-Metabol W8 technique. Fastness grades on cotton include—Light 5, washing (repeated severe, five times at 100°C.) 3, hypochlorite 4.

**PROPOLAS YELLOW 3 GS**—This is a level-dyeing acid dye which gives dyeings having higher fastness to wet treatments than the ordinary level-dyeing acid dyes. It is of particular interest on cloth, especially carbonised materials, in conjunction with Solway Rubanol R, Blue 2 G, and Celestol B. It has high affinity for nylon from weakly acid dyebaths, and is suitable for producing deep dyeings on wool-nylon mixtures. It may be printed on wool, silk (unweighted and tin-weighted), and nylon. Fastness figures on wool include—Light 6-7, washing (repeated mild, five times at 40°C.) 3, perspiration 4.

**POLYMON VIOLET R**—This is a modified form of the colour lake Deep Amaranth 15515, introduced for the mass coloration of plastics and giving colourings similar in hue and properties to those obtained with the older product but much more readily dispersible. It gives reddish violets having good transparency, good heat stability, and moderate light fastness in opaque reduced shades. It is mainly of interest for colouring thermoplastics such as cellulose acetate and nitrate, polystyrene, and polythene.

**THE BLEACHING OF JUTE FOR TEXTILE PURPOSES**—This Technical Circular is a reprint of a paper by B. P. Ridge and A. H. Little originally published in *J. Textile Inst.*, 33, T 33 (1942).

**INVESTIGATIONS INTO THE DYEING OF SYNTHETIC FIBRES**—This Technical Circular is a reprint of a paper given by T. Vickerstaff to the Institut Textile de France on 17th October 1950 (cf. *J.B.D.C.*, 67, 353 (1951)).

**THE DYEING OF ANODISED ALUMINIUM WITH ALIMAX DYES**—This Technical Circular contains information designed to assist the anodiser in choosing suitable dyes and dyeing methods for colouring anodised aluminium. A brief description is given of the preparation of the film, including the three best known commercial anodising techniques, and of the normal (high-temperature) and alternative cold dyeing methods. The effect of alloying constituents on the hue and the fastness of dyeings produced with the Alimax and selected Ultralan dyes is discussed and also the production of multicoloured and patterned effects using resist processes, and direct printing using stencils or screens. Fastness data are given for nineteen dyes, including fastness to light, heat (including prolonged heating), cold and boiling water, and washing. In each case figures are given in respect of pale and full dyeings on sulphuric acid films and of full dyeings only on chromic acid films.

**FIXANOL P: APPLICATION IN TEXTILE PRINTING**—This Technical Circular contains light and washing fastness data for a wide range of prints produced with and without the use of Fixanol P. Table I gives figures for the fastness of

prints produced using a range of acid dyes on viscose rayon by the urea method. One set of figures is for prints washed off in water only, the other for prints washed off in a 0.1% soln. of Fixanol P. Table II contains similar data for a range of direct dyes. Table III contains comparative figures for prints using acid dyes washed off in cold water and then aftertreated in a solution of Fixanol P at 70°C. Table IV is like Table III, but for a range of direct dyes. In discharge printing it has been shown that either pretreatment before discharging or addition of Fixanol P to the first wash water produces inferior results.

**VELAN PF: ITS EFFECT ON DYED COTTON MATERIAL**—This Technical Circular gives figures for the fastness to light and the effect on colour of dyings of a very extensive range of dyes when these are aftertreated with Velan PF (a) by using 5% of the reagent to give a water-repellent finish, and (b) by padding the material in a 0.2% soln. at 80% mangle expression to give a soft finish. Classes of dyes examined include direct and aftertreated direct dyes; sulphur dyes, including the Thionol M brands; vat dyes, including the solubilised types; and azone combinations.

**LANOC CN: A SUBSTANTIVE, DURABLE RUBBERPROOFING AGENT**—This Technical Circular is prefaced by a brief account of the life cycle of the common clothes moth. A general account of the properties of Lanoc CN is then given, followed by details of methods of application, including use in conjunction with Ultralan dyes, application to peroxide-bleached wool, application to undyed goods, and by the Carbolan Salt A method with Glauber's salt only. Rates of exhaustion of Lanoc CN on to wool are illustrated graphically for (a) a sulphuric acid bath at different temperatures, (b) different dyebath conditions but with regular increase of temperature, (c) different dyebath conditions, and (d) a neutral dyebath with regular temperature increase and in the presence of Carbolan Salt A. A final graph illustrates the rate of transfer of Lanoc CN from treated to untreated material.

#### Sandoz Products Ltd.

**RESOFIX DYES: DYES ON COTTON**—This card shows a range of seventeen dyes. The Resofix dyes are direct dyes selected to give, when aftertreated on the fibre with a solution containing ammonium copper sulphate and Resofix VF, dyings having wet fastness properties superior to those given by the Cuprofix range. Sample dyings are appended in two depths with and without aftertreatment. Also alongside are shown samples composed of dyed or dyed-aftertreated yarn and undyed yarn before washing and after washing for 60 min. in a solution containing 0.5% of soap and 0.2% of soda ash at (a) 60°C. and (b) 90°C. A further range of six pairs of patterns is shown dyed to approximately the same hue with vat dyes and Resofix dyes, while opposite the relative cost of using each type is illustrated graphically.

**SOLAR RED BROWN 2 RL**—This class B direct dye gives dyings, of excellent fastness to light, which are unaffected by crease resist finishing. Solid dyings are produced on cotton viscose rayon materials, and it is also of interest for filling in the cotton in wool-cotton unions, when the addition of Thionat RS enables a very good reserve to be obtained on the wool. Fastness figures include—Light 7, washing at 40°C. 3, perspiration 2-3.

**SOLAR BROWN RLN**—This direct dye gives reddish browns on all types of cellulose materials, and is recommended for use alone and in mixtures for dyings of high light fastness. It is a class B dye, and is suitable for use in circulating liquor machines and for padding. Solid dyings can be obtained on cotton-viscose rayon mixtures; acetate rayon is reserved. Fastness figures on cotton include—Light 3-6, washing at 40°C. (light shades) 4, perspiration 3-4.

**SULPHONATE GREY BWL**—This neutral-dyeing acid dye is very suitable for dyeing loose wool, slubbing, and yarn when fastness to severe milling is required. It is unaffected by the presence of chrome in the dyebath, and the hue of the dyeing is practically unaltered in the presence of copper and iron. It is also of interest for dyeing nylon, wool-nylon mixtures, and silk. Fastness figures include—Light 6-7, alkaline milling 5, perspiration 5.

**XYLENE FAST RED 2 GP**—This acid dye is characterised by good level-dyeing properties and moderate fastness to wet treatments on wool, so that it is very suitable

for use alone or in mixtures on hosiery yarn and fabric. Dyings have good fastness to steaming, hot pressing, and decatizing. It is also of interest for dyeing silk. It is not subject to sulphite fault formation.

**BRILLIANT ALIZARINE MILLING BLUE FGL**—This acid dye is recommended for use alone or in mixtures on wool. It gives bright blues, brighter and slightly redder than when Brilliant Alizarine Milling Blue G is used, and considerably greener and still a little brighter than with the BL brand. It is suitable for shading chrome dyes and is also of interest on nylon and silk. It is not suitable for printing. Fastness figures on wool include—Light 6, washing at 40°C. 4-5, perspiration 5.

**BRILLIANT ALIZARINE SKY BLUE 2 GB**—This acid dye is recommended for application from weakly acid or neutral dyebaths to give greenish blues of good fastness to light and wet treatments. It is useful for brightening chrome dyings, particularly when single-bath methods are employed. On silk brilliant shades of good fastness are obtained, and it has high affinity for nylon. Fastness figures on wool include—Light 6, washing at 40°C. 4-5, water 4-5.

**CHLORAMINE BRILLIANT GREENS BN**—This direct dye gives bright bluish greens, of good fastness to water and moderate fastness to washing, which are not significantly affected by crease resist finishing. Solid dyings may be obtained on cotton-viscose rayon mixtures, while nylon and acetate rayon are reserved white, the latter when using Type ACS. It is also suitable for rubberproofing if Type Vulco is employed. Fastness figures on cotton include—Light 3, perspiration 3, water 4.

**CUPROFIX BROWN CBL, C3BL, AND CRL; CUPROFIX YELLOW BROWN CRGL**—These direct dyes give dyings of very good fastness to light, washing, and wet treatments generally when aftertreated with Cuprofix 8 or copper sulphate, and are suitable for use on all cellulose material which do not have to withstand boiling or hypochlorite bleaching. They may be used for dischargeable grounds on cotton and rayon furnishings and dress goods. Each is shown as dyings on cotton and spun viscose cloth, on cotton and continuous-filament viscose yarn, and with white and coloured discharge screen prints, aftertreated in each case with either Cuprofix 8 or copper sulphate.

**CUPROFIX NAVY CBL, CFSL, AND CSL; CUPROFIX BLUE CBL**—This card shows four dyes from the Cuprofix range similar in dyeing properties and uses to those described in the preceding card.

**DIAMAZINE FAST BROWN RL; 3 GL**—These direct dyes give full, dark browns on cotton and cotton-viscose rayon mixtures when diazotised and developed on the fibre with  $\beta$ -naphthol. Dyings have good fastness to wet treatments; may be discharged to white under both neutral and alkaline conditions; are suitable for crease-resist finishing, the fastness to light being improved; and may be rubberproofed if Type Vulco is used. Fastness figures on cotton for dyings both of the Brown RL and the Brown 3 GL diazotised and developed with  $\beta$ -naphthol include—Light 3, water 4-5, perspiration 4-5.

**DIAMAZINE FAST ORANGE RL**—This direct dye yields yellowish oranges with Yellow Developer C and reddish oranges with  $\beta$ -naphthol when diazotised and developed on the fibre. Its dyeing characteristics and uses are similar to those of the Diamazine Browns RL and 3 GL described in the preceding card. Fastness figures on cotton include—

Aftertreated with—	Light	Water	Perspiration
Yellow Developer C	5-6	4-5	4-5
$\beta$ -Naphthol	4	4-5	4-5

**DIAMAZINE FAST YELLOW GL**—This direct dye is recommended for use alone and in mixtures, particularly with Diamazine Fast Green 2 GL and GLN, to give dyings which when aftertreated with diazotisation and development with Yellow Developer C have good fastness to light, water, washing, and perspiration. It is dischargeable to white under both alkaline and neutral conditions, will give solid dyings on cotton-viscose rayon mixtures, and withstands crease-resist finishing and rubberising if Type Vulco is employed. Fastness figures on cotton for a diazotised and developed dyeing include—Light 4, water 4-5, perspiration 4.

**GRAPHOL FAST BLUE BLP PASTE ON PAPER**—This is an extremely fine aqueous dispersion of Graphol Fast Blue BL powder which does not require grinding with the pulp in the beater but may be added in the later stages of beating without loss in depth. Prolonged storage does not affect the dyeing strength of the paste, which may be diluted with cold water to any degree to give mixings which remain stable for a considerable time. It is specially recommended for bleuing bank note and high-grade writing papers. It is not suitable for dyeing unsized papers. Fastness figures include—Light 7-8, water 5, acids 5, alkalis 5.

### Yorkshire Dyeware & Chemical Co. Ltd.

**DYE STUFFS FOR LEATHER**—This card describes a range of fifty-four dyes, which are shown on full chrome side leather, full chrome suede split, and Persian sheepskin, where considered suitable. Figures for solubility, penetration, and fastness to light are appended, together with details of the level-dyeing properties and the suitability of each dye for brush dyeing. The text contains full details of the dyeing procedures recommended, including a method for applying Hematine Crysta<sup>l</sup> No. 2 Cone, and also for producing a black by diazotisation and development with *m*-toluenediamine.

## Abstracts from British and Foreign Journals and Patents

(The titles of abstracts may be modified. Abbreviations of names of firms are listed in I.S.D.C., 68, 23 (Jan. 1952), while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)

### I—PLANT; MACHINERY; BUILDINGS

#### PATENTS

**Solvent Degreasing of Raw Wool.** Smith, Drumm & Co. *USP* 2,593,422

**Reinforced Covering for Textile-squeezing Rollers.** G. H. Bottomley. *BP* 680,446

Textile-squeezing rollers are covered in the usual manner with a spiral wrapping of top sliver, which is then reinforced with an outer skeleton of relatively strong yarn, e.g. nylon, which can be wound criss-cross, and which leaves enough exposed top to give the usual sprung absorbent surface. J. W. B.

**Wet Processing of Filamentary Materials of Indefinite Length.** American Viscose Corp. *USP* 2,593,555

Yarn, sliver, or tow, etc. is advanced (by means of a reel or two or more canted rollers or drums) through a path comprising several laterally displaced convolutions about one or more cylinders. At least one of the cylinders has a liquid-permeable outer surface or covering, upon which the yarn, etc. is carried and through which liquor may be supplied to the yarn in any suitable fashion. C. O. C.

**Yarn-scouring Machine.** J. Dean. *BP* 679,053

Compensating means are provided to take up the slack in the top tapes caused by their becoming dry, and further compensating means provide even tension on each tape (both top and bottom sets) independently. C. O. C.

**Impregnating Yarn.** Dan River Mills. *USP* 2,592,632

A driven dip roll is partly immersed in the liquor. There are also two rolls freely rotatable on axes parallel to the dip roll. One of these rolls has a resilient surface in rolling contact with the dip roll and the other has spaced peripheral grooves increasing gradually in depth from end to end. The dip roll is driven in a direction whereby a series of successive loops of yarn starting at the shallowest of the grooves are passed against the dip roll in varying states of decreasing tension to the deepest of the grooves. Better and more uniform impregnation is given. C. O. C.

**Pressure Dyeing, etc. Machines.** Groeninghe Vervenij P.V.B.A. *BP* 678,952 *USP* 2,592,687

Static pressure is applied to the liquor through an expansion chamber having a level gauge and at both ends a tap. This chamber is branched on to the suction conduit of the pump. By having the expansion vessel never completely full nor ever entirely empty during processing, an air chamber is provided which exerts resilient pressure on the circulation apparatus and on the material to be treated. If compressed air is used to produce the static pressure, it is introduced through the upper part of the expansion vessel, and, because of visual control by the level gauge, no air penetrates the suction conduit of the pump. Where the pressure is produced by heating the bath, the air in the upper part of the expansion chamber is compressed, and damage to the plant is avoided. C. O. C.

**Drum Washing Machine.** Murray Corp. *USP* 2,593,752

Washing, rinsing, and hydroextraction are carried out

automatically. The liquor is positively forced through the material being treated. C. O. C.

**Wet Treatment of Textiles, Clothing, Plastics, etc.** P. E. Whittington. *USP* 2,593,640

A machine is described in which the treatment can be carried out under a partial vacuum and at controlled temperature. C. O. C.

**Controlling Supply of Liquid to Washing or Drying-cleaning Machines.** Isaac Braithwaite & Son Engineers. *BP* 681,048

In washing, etc. machines in which two liquids are supplied through a pump connected to the two sources of supply, flow of liquid from one system to another, e.g. from hot water to cold water or vice versa, and through the pump is prevented when the pump is stopped, but flow of liquid from both systems automatically allowed when the pump starts up. C. O. C.

**Tenter.** J. Dungler. *BP* 680,125

**Blowing or Decatising Machines.** William Whiteley & Sons. *BP* 678,949

Means are described which render it impossible for the clutches controlling the driving of the two steaming rollers to be engaged at the same time. C. O. C.

**Nylon-preboarding Machine.** Grimsley & Co., Leicester. *BP* 680,636

A vertical, rectangular wall mounted on a rotary table carries on each face a set of metal hosiery forms. The treating chamber, normally open at one side, and mounted on rails, travels forward and is closed by the wall during the steaming period. Retraction of the chamber enables the wall to be turned for changing the hose on the forms. An electrical timing control initiates the various operations. G. E. K.

**Coating by Electrostatic Deposition.** Harper J. Lansburg Co. *BP* 679,814

Dielectric particles coated on a material, e.g. by electrostatic deposition, which are so far apart that normally they would not spread and flow together to form a continuous film, are caused to coalesce into a thin film by treatment in an electrostatic field. C. O. C.

**Coating or Printing by Electrostatic Precipitation.** W. C. Huebner. *BP* 679,974

The material to be coated or printed is passed through an electrostatic field while in contact with smoke or similar gaseous suspension of finely divided solids so as to cause the solids to migrate and deposit upon it. C. O. C.

**Cutting.** Samson Holding Corp. *BP* 680,312

Production of a pile of uniform layers of fabric is described. C. O. C.

**Winding Flexible Sheet Material.** Timsons. *BP* 680,331

A friction clutch in the drive comprises two friction elements adapted to be slipped to a variable degree as required to reduce the tension on the material. They are controlled by a member responsive to the tension of the running material. C. O. C.

**Shrinking Fabrics** (X p. 536).

### III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

**Complexones. XXIII—Phenoxy Oxygen as Co-ordination Partner.** G. Schwarzenbach, G. Anderogg, and R. Sallmann. *Heile. Chim. Acta*, **35**, 1785-1793 (Oct. 1952).

Phenol deriv. having  $\text{CH}_3\text{N}(\text{CH}_3)\text{COOH}_3$  *ortho* to the OH group are found to form metal complexes even with Mg and the alkaline earth metals, Ca, Ba, and Sr. The stability of these complexes is similar in magnitude to the stability of the complexes of nitrotriacetic acid.

H. H. H.

**Saponification of Wool Wax and Recovery of the Wax Alcohols.** C. S. Barnes, R. G. Curtiss, and H. H. Hatt. *Australian J. Appl. Sci.*, **3**, 88 (1952); *J. Amer. Leather Chem. Assoc.*, **47**, 493 (July 1952).

Wool wax is rapidly and completely saponified by either NaOH or KOH in aqueous ethanol. The products are separated by extracting the wax alcohols from the wax soaps and aqueous alcoholic liquors by an immiscible solvent, such as benzene. The process is suitable for large-scale operation.

C. J. W. H.

**Mechanisms of Reactions for introducing Sulphonic Acid Groups into Fatty Acids.** J. H. de Boer. *Rec. Trav. chim.*, **71**, 814-821 (Aug. 1952).

A review of the work by H. J. Baecker and his collaborators on the introduction of sulphy groups into fatty acids is given, and mechanisms are postulated for the reactions. The  $\text{SO}_4^{2-}$  ion is thought to be the agent in direct sulphonation, whereas  $\text{HSO}_3^-$  may be active in the addition of bisulphite to an  $\alpha, \beta$  double bond in an unsaturated fatty acid. The  $\text{SO}_3\text{H}$  radical may be responsible for the substitution of halogen atoms by  $\text{SO}_3\text{H}$  groups in fatty acids.

C. H. R.

**Behaviour of Sodium Chlorite at the Dropping Mercury Cathode.** N. Konopik. *Sitzungsber. Öster. Akad. Wiss.*, **161**, (1-2), 255-257 (1952).

A Degussa preparation of sodium chlorite ( $\text{NaClO}_2$ ), when investigated polarographically in  $\text{NaOH}$  as primary electrolyte against the standard calomel electrode, gives a pronounced step at  $\sim 1.0 \text{ V}$ , which exhibits several peculiarities—no linear relationship in  $\text{NaOH}$ , although the step height is a reproducible function of the chlorite concn., and still appears with  $10^{-6} \text{ M NaOH}$  soln. The half-wave potential is displaced at the lower chlorite concn. to positive values. Compared with the equivalent step for lead in  $\text{NaOH}$ , the step height corresponds to a one-electron reduction. The Ilková equation is valid, and the reaction must be regarded as irreversible. Chloride and chlorate ions are without influence, but the  $[\text{H}^+]$  affects the step height, which has a pronounced maximum between pH 14 and 15, with a steep decline at either side. No step appears in  $\text{LiOH}$  or  $\text{Ba}(\text{OH})_2$ , and continued addition of Ca or Mg ions to  $\text{NaOH}$  causes it to disappear. The wave form is also very characteristic.

H. H. H.

**Application of Peroxy Compounds in the Textile Industry.** R. A. Secord. *Amer. Dyestuff Rep.*, **41**, P. 584 (15 Sept. 1952).

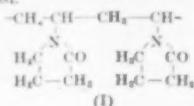
A review of the properties, relative value, and application (in bleaching, and oxidation of vat dyes) of  $\text{H}_2\text{O}_2$ ,  $\text{Na}_2\text{O}_2$ ,  $\text{NaBO}_3\text{H}_2\text{O}_2$ ,  $\text{H}_2\text{O}_2\text{H}_3\text{O}_2\text{O}_2$ , and  $\text{NaBO}_3\text{H}_2\text{O}_2\text{H}_3\text{O}_2$ .

J. W. D.

**Polymerisation of N-Vinylpyrrolidone.** J. W. Breitenbach and A. Schmidt. *Sitzungsber. Öster. Akad. Wiss.*, **161**, (5), 833-834 (1952).

Poly N-vinylpyrrolidone are of especial interest, since they constitute a transition class between the macromolecular polymers and the polypeptides. The macromolecular chain is entirely  $\text{C}-\text{C}$ , with a lactam ring to every chain unit (1). The kinetics of formation shows that—(1) pure liquid N-vinylpyrrolidone has only a slight tendency to polymerise, e.g. 0.026% hr. at 140°C., but at higher temp., e.g. 180°C., it is 0.60% hr. (2) At lower temp., polymerisation is effected by peroxides and aliphatic azo compounds. (3) The average degree of polymerisation depends only slightly on the concn. of the azo catalyst (e.g. *a*-azobisisobutyronitrile). (4) Temp. dependence of the average degree of polymerisation is small. (5) N-Vinyl-

pyrrolidone when treated with phosphorus pentoxide is slowly dimerised.



H. H. H.

**Technical Applications of Gluconic Acid.** P. Wengraf. *Textil Rund.*, **7**, 319-323 (July 1952).

The properties of gluconic acid are detailed and potential textile applications outlined. It is a weak acid suitable for use in dyebaths and in resin precondensates. Mg and Ca complexes are formed above pH 12. Heavy-metal salts may find applications in water-repellent and fireproofing finishes.

G. L.

## PATENTS

**Detergent.** Monsanto. *U.S.P. 2,594,257-8*

Addition of attapulgite (a complex hydrated magnesium aluminum silicate occurring naturally as specular particles) to a mixture of ethylene oxide-thiol condensate and inorganic detergents results in lower soil redeposition during washing and a softer handle in the cleansed fabric.

C. O. C.

**Detergents resistant to Hard Water.** Monsanto. *U.S.P. 2,594,421*

The condensates of 5-20 mol. of ethylene oxide and 1 mol. of an aliphatic nitrile are highly efficient non-ionic detergents resistant to hard water.

U.S.P. 2,594,431

A mixture of the condensate of abietic acid or a derivative thereof (1 part by wt.) with ethylene oxide (0.5-2.3), tetrasodium pyrophosphate and/or sodium tripolyphosphate and/or trisodium phosphate and/or sodium hexametaphosphate (20-50), and starch and/or carboxymethyl cellulose (to make 100) is an efficient detergent having reduced tendency to foam and stable to alkali.

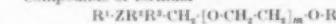
U.S.P. 2,594,453

A mixture of the condensate (15-30 parts by wt.) of tall oil (1) with ethylene oxide (0.5-2.3), one or more of the above phosphates (20-50),  $\text{Na}_2\text{CO}_3$  and/or  $\text{Na}_2\text{SiO}_3$  and/or  $\text{Na}_2\text{SO}_4$  and/or  $\text{Na}_2\text{borate}$  (20-40), and carboxymethyl cellulose (0.1-5.0) is a good non-foaming detergent.

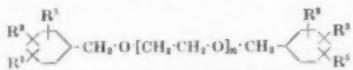
C. O. C.

**Non-ionic Surface-active Agents.** Rohm & Haas Co. *U.S.P. 2,596,091-3*

Compounds of formula—



or

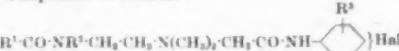


( $\text{R}^1$  = Alk of  $> 5 \text{ C}$ ;  $\text{R}^2$  = H or  $\text{CH}_3$ ;  $\text{R}^4$  = H or  $\text{R}^1$  and of  $< 5 \text{ C}$ ;  $\text{R}^5$  = Alk of 6-15 C;  $m$  = 5;  $n$  = 12-50; Z = a benzoid or naphthaloid radical) are non-ionic surface-active agents which are unaffected by the presence of large amounts of strong alkali.

C. O. C.

**Bactericidal Wetting and Detergent Agents.** Gy. BP 680,476

Compounds of formula—



( $\text{R}^1$  = Alk of 7-15 C;  $\text{R}^2$  = lower Alk;  $\text{R}^3$  = H and/or Br and/or Cl and/or  $\text{CH}_3$  and/or  $\text{C}_2\text{H}_5$ ) are mostly crystalline solids with strong bactericidal, wetting, and detergent properties.

C. O. C.

**Softening and Preparing Agents for Leather and Textiles.** Baaf. *BP 680,588*

Compounds of formula—



( $\text{R}^1$  and  $\text{R}^2$  = alkylene of 2-6 C) are good softening and preparing agents. Their solubility or dispersibility in water is improved if they are condensed with fatty acid hydroxyalkylamides.

C. O. C.

**Self-dispersible Hydroxymethylamide Water-repellent Compositions.** General Aniline.

BP 679,811

Compounds of formula  $R^1-CO-NR^2-CH_2OH$  ( $R^1$  = a radical containing at least one aliphatic or cycloaliphatic residue of  $> 3$  C;  $R^2$  = H or an aliphatic radical) are obtained as dry solids which disperse readily in water by melting them and then dissolving in them a suitable dispersing agent.

C. O. C.

**Waterproof and Laundry-proof Fabrics coated with Paraffin Wax.** K. A. Fischer. USP 2,594,547

The neutral material of m.p.  $> 100$  °C. obtained from the hydrogenation of CO adheres tenaciously to fabrics, strengthening them and imparting dimensional stability. It may be applied to fabrics either in the molten state or from solution in an organic solvent. The treated fabrics are waterproof.

C. O. C.

**Carbalkoxyhydroxybenzocoumarins as Fungicides.**

Schaefflein & Co. USP 2,596,107  
3-Carbalkoxy-4-hydroxybenzocoumarins and 3-carbalkoxy-4-hydroxyhydrobenzocoumarins, e.g.—



C. O. C.

are very effective fungicides.

**Acrylonitrile Protein Derivatives.** U.S. Secretary of Agriculture. USP 2,594,293

Treatment of natural proteins, e.g. zein and casein, with acrylonitrile in presence of alkali yields products which retain all the desirable characteristics of the natural proteins but which in aqueous alkaline dispersion have a markedly decreased tendency to gel in presence of formaldehyde. Products, e.g. fibres, paper coatings, water paints, or leather dressings, formed from such substances have markedly increased water resistance compared with those prepared from natural proteins.

C. O. C.

**Cellulose Ester and Ether Emulsions.** H. Grunwald. BP 679,764

Water-in-oil emulsions of cellulose derivatives are obtained by first preparing a "water-in-oil" emulsion of a solvent or a solvent mixture and then adding the shredded cellulose derivative, which will readily dissolve and simultaneously disperse in the continuous phase. They can be applied by spreading with knife-blade or roller, dipping, or rubbing, and give coatings which because of their penetration into textiles, etc. are extremely resistant to water, dirt, and grease.

W. G. C.

Reaction of Cellulose with Formaldehyde (XI p. 537).

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

**Phenols by Dehydrogenation.** H. C. Chitwood, J. T. Fitzpatrick, G. W. Fowler, and B. T. Freure. *Ind. Eng. Chem.*, **44**, 1696-1698 (July 1952).

Phenol was prepared in 15% yield by the dehydrogenation of cyclohexanol and cyclohexanone at 350 °C. using a catalyst made from nickel, copper, chromium, platinum, and alkali-metal sulphate on an inert support. The catalyst maintained its activity for  $> 100$  hr. operation, and was fully regenerated by oxidation and reduction. Substituted phenols were prepared similarly in excellent yield from the corresponding substituted cyclohexanol and cyclohexanone.

W. K. R.

**Metal Indicators. VI—A Nitrophenol forming Complexes with Metal Cations.** G. Schwarzenbach, C. Anderegg, and R. Sallmann. *Helv. Chim. Acta*, **35**, 1794-1796 (Oct. 1952).

A deriv. of *p*-nitrophenol having a  $-\text{CH}_2-\text{N}(\text{CH}_2-\text{COOH})_2$  group *ortho* to the OH changes its light absorption when metal cations are added. Increasing shift of the absorption band to shorter wavelengths has been observed in the series  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{H}^+$ ,  $\text{Mg}^{2+}$ , and  $\text{Zn}^{2+}$ , which is due to increasing electron-sharing ability of the cations. The

proton of this series is held primarily by the bisacetoxy-methylamino group and forms a hydrogen bond between the N atom and the O of the phenolic OH. A proton held primarily by the phenoxide oxygen has a stronger influence than  $\text{Zn}^{2+}$  and other "heavy" metal cations. H. H. H.

**Preparation of 2,6-Dinitrophenol.** M. A. Phillips. *Chem. and Ind.*, 714-715 (19th July 1952).

Nitration of 4-hydroxy-3-nitrophenylarsonic acid (commercial product) in monohydrate at room temp. gave a 90% yield of 4-hydroxy-3,5-dinitrophenylarsonic acid, which on refluxing for 4.5 hr. with sodium bisulphite gave 2,6-dinitrophenol in 91.5% yield.

C. H. R.

**Proof of the Constitution of 2-*tert*-Butyl-6-methylbenzoic Acid.** P. J. C. Fiers and J. van Rysselberghe. *Bull. Soc. chim. Belg.*, **61**, 215-222 (June-July 1952).

2-*tert*-Butyl-6-methylaniline was obtained by heating *o*-toluidine with *tert*-butyl alcohol in the presence of zinc chloride. This with formic acid gave the formamide, which was reduced by zinc dust to 2-*tert*-butyl-6-methylbenzonitrile. Treatment of this with sulphuric acid gave the benzamide, which was converted to the benzoic acid (I) by hydrolysis with phosphoric acid. Oxidation of I with nitric acid gave a tricarboxylic acid which was identical with that obtained by oxidising 2,3-dimethylbenzoic acid with  $\text{KMnO}_4$ .

C. H. R.

**Reactions with Dry Alkaline-earth Hydroxides.**

**II—Reductions of 1-Nitro- and 1,5-Dinitronaphthalene.** J. Datta. *J. Indian Chem. Soc.*, **29**, 394-396 (June 1952).

Dry distillation of 1-nitronaphthalene with sulphur and calcium hydroxide gave the corresponding naphthylamine, of which  $> 50\%$  decomposed to give ammonia and naphthalene. 1,5-Dinitronaphthalene under similar conditions decomposed completely to give a quantitative yield of ammonia.

C. H. R.

**Trityl Derivatives of Amines. I—*N*-Triphenylmethyl Derivatives of Aromatic Primary Amines.** P. E. Verkade, H. Nijon, F. D. Tollenaar, J. H. van Rij, and M. van Leeuwen. *Rec. Trav. chim.*, **71**, 1007-1011 (Sept.-Oct. 1952).

Equimolar quantities of a monoamine and trityl chloride  $(\text{C}_6\text{H}_5)_3\text{C-Cl}$  react at room temp. in the presence of excess pyridine to give *N*-trityl deriv. in yields usually  $> 90\%$ . Under the same conditions, and using the appropriate amount of trityl chloride, diamines, etc. yield bis-*N*-trityl deriv., etc. The amine must not contain two substituents *ortho* to the  $\text{NH}_2$  group. 2,4,6-Triptyl deriv. are described. The sharp m.p., which are often widely divergent with isomers, render the trityl deriv. suitable for identification purposes.

H. H. H.

**Preparation of 8-Naphthylamine-1:3:5-trisulphonic Acid (K Acid), and Identification of the Side Products.** F. Allison, G. Brunner, and H. E. Fierz-David. *Helv. Chim. Acta*, **35**, 2139-2144 (Oct. 1952).

The preparation of 8-naphthylamine-1,3,5-trisulphonic acid by a modified method is described, whereby an improvement in yield is recorded over that given by the standard process. It is based on reduction of the 8-nitro-1,3,5-trisulphonic acid, which can be readily isolated as the pure potassium salt. Of the reagents, ammonium sulphide in ammonia solution is preferable to acid reducing agents, or to reduction by hydrogen in neutral solution with nickel catalyst. The latter produces some free ammonia. It is also found that 5.5% of 1-hydroxy-naphthalene-2,4-dinitro-6,8-disulphonic acid is formed during the nitration of naphthalene-1,3,5-trisulphonic acid.

H. H. H.

**Preparation of 3-Diazo-2-hydroxy-1-methylbenzene-5-sulphonic Acid.** K. H. Klaasens and C. J. Schoot. *Rec. Trav. chim.*, **71**, 920-924 (August 1952).

Sulphonation of *o*-cresol with sulphuric acid at 110 °C. gave 85% yield of the 5-sulphonic acid, which on nitration with  $\text{KNO}_3$  or  $\text{HNO}_3$  gave 3-nitro-2-cresol 5-sulphonic acid. This was reduced to the amino compound electrolytically, with sodium sulphide, and with Fe and HCl. The highest yield of the crystalline Na salt of 3-diazo-2-hydroxy-1-methylbenzene-5-sulphonic acid was obtained by adding a few drops of conc. fluoroboric acid to a slimes suspension of the amine in aqueous acetone, and then adding  $\text{NaNO}_2$  soln.

C. H. R.

**Kinetics and Mechanism of Diazotisation—IV.** H. Schmid and A. Woppmann. *Sitzungsber. Öster. Akad. Wiss.*, **161**, (3-4), 346-364 (1952).

The kinetics of the diazotisation of aniline in nitric acid has been investigated up to an acid concn. of 0.2 M. at 0°C., and the results establish that—(1) The order of the nitrous acid in the reaction equation is 2, that of the anidinium ion rises from 0 to 1 with increasing acid concn., and that of the hydrogen falls with increasing acid from 0 to 1. (2) The upper limit of the velocity equation at high acid concn. is that previously found by Schmid and Muhr (cf. *Ber.*, **70**, 421 (1937)) for the diazotisation of aniline at high  $H_2SO_4$  concn. (0.1-0.2 M.)

$$\frac{d[C_6H_5N_2^+]}{dt} = k_1 [C_6H_5NH_3^+] [HNO_2^+] \\ - k_2 [H_2O^+]^2$$

(3) The lower limit is that reported by Hughes, Ingold, and Ridd (*Nature*, **166**, 642 (1950)) for the diazotisation of aniline perchlorates in buffer solutions—

$$\frac{d[C_6H_5N_2^+]}{dt} = k_3 [HNO_2^+]^2$$

(4) The general time law over the total acid range is—

$$\frac{d[C_6H_5N_2^+]}{dt} = k_1 [HNO_2^+]^2 [C_6H_5NH_3^+] \\ - k_2 [H_2O^+]^2 + [C_6H_5NH_3^+]$$

at 0°C.  $k_1 = 55$  and  $k_2 = 0.06$ . The temperature coefficient for  $k = k_1/k_2$  is 1.8-10°C. (5) The general velocity equation indicates a reaction mechanism involving the nitrosation of free aniline by nitrous anhydride.

The reaction mechanism of catalysis by chloride and bromide ions is discussed in connection with the present work, and the general scheme for aromatic diazotisation shown to include aliphatic amines and the relevant parallel decompositions of ammonium salts. Finally, the rate of diazotisation of aniline in HCl is formulated as—

$$\frac{d[C_6H_5N_2^+]}{dt} = k_4 [HNO_2^+] \\ [C_6H_5NH_3^+] [HNO_3] \left( k_5 [H_2O^+] + [C_6H_5NH_3^+] + k_6 [Cl^-] \right)$$

There are 18 tables of data and 41 references. H. H. H.

**New Synthesis based on 5-Aminopyrimidines.** F. L. Rose. *J.C.S.*, **3448** 3484 (Sept. 1952).

A series of 5-aminopyrimidines has been prepared, and the similarity of the behaviour of the 5-amino group to that in aromatic primary amines is demonstrated. Although the 5-amino group can be diazotised, the presence of a 4(6)-mercapto-, hydroxyl, or primary amino group leads to the rapid formation, even under acid conditions, of pyrimidino-thiadiazoles, -oxadiazoles, and -triazoles respectively. Ring closure also occurs, most readily in the presence of caustic alkali, when alkyl groups are present in positions 4 and 6, and the products are 1,2,4,6-tetra-azadiones, isomeric with the purines. The preparation of many homologues and analogues is described. The 5-amino group exhibits normal behaviour towards acylating agents, and also in its reaction with diacyanamide and diacyanamides, from which a series of pyrimidyl-dguanides is prepared. H. H. H.

**Solvent Effects in *cis-trans* Isomerisation.** P. Lunet and C. A. Winkler. *Canadian J. Chem.*, **30**, 679-686 (Sept. 1952).

The *cis-trans* isomerisation of azoxybenzene and 4,4'-azoxytoluene has been studied in various solvents. A linear relationship exists between  $\log k$  ( $k$  = rate constant) and  $P_L + P_L^2$  (internal pressure of the solvent) when  $P_L > 9$ . Linear relationships have also been observed between  $\log k$  for azobenzene, azoxybenzene, and 4,4'-azoxytoluene in a series of solvents. In the case of *p*-chlorobenzenediazoncyanide, the above relationship did not hold. Decomposition of the *cis* form of this diazoncyanide took place in nonpolar solvents, whereas the *trans* form was stable under similar conditions. C. H. R.

**Polarographic Reduction of *cis*- and *trans*-Azo-benzene.** P. J. Hillson and P. P. Birnbaum. *Trans. Faraday Soc.*, **48**, 478-482 (May 1952).

The polarographic reduction potentials of *cis*- and *trans*-azobenzene are the same in acid soln., whereas they differ markedly in alkaline soln. Since this difference depends on pH, it does not measure the difference in free energy between the two forms. The similar behaviour of maleic and fumaric acids suggests that this phenomenon

is common to the polarographic reduction of double-bonded compounds capable of *cis-trans* isomerism. Reaction is considered to take place in two steps, of which the first, occurring at the electrode, is accelerated at low pH; the metastable *cis* form is then converted into *trans*, more rapidly than the net rate of reduction. The occurrence of this reaction at a mercury surface has been demonstrated directly. J. W. D.

**Possible Occurrence of Geometrical Isomerism among the Diazosulphones.** H. C. Freeman, R. J. W. Le Feuvre, J. Northcott, and I. Youhovsky. *J.C.S.*, **3381** 3384 (Sept. 1952).

From an examination of the dipole moments and spectra of  $C_6H_5N\cdot SO_2C_6H_5$  and five of its derivs., the azo group appears to be *trans* in the known forms. Irradiation of their soln. in benzene or alcohol, however, provides signs of a reversible change beneath an irreversible photodecomposition. H. H. H.

**Polarities and Ultraviolet Spectra of Phenyl *p*-Tolyl Azoxysulphone and its Dialkylamino Derivatives.** H. C. Freeman, R. J. W. Le Feuvre, J. Northcott, and C. V. Worth. *J.C.S.*, **3384** 3389 (Sept. 1952).

The dipole moments of phenyl *p*-tolyl azoxysulphone and of its *p*-dimethylamino and *p*-diethylamino deriv. are 6.0 D., 8.5 D., and 8.9 D., respectively. These polarity changes can be explained by mesomerism. The spectra between 2300 and 4700 Å. of all three substances in ethanol are recorded, and the spectrum of phenyl *p*-tolyl azoxysulphone resembles that of azoxybenzenes in the 3000 Å. region. No indications of *cis-trans* isomerism have been detected by the usual tests. H. H. H.

**Azo Dyes from Formaldimethone.** B. H. Iyer and M. V. Kowalg. *J. Indian Inst. Sci.*, **34**, 81-86 (1952); *Chem. Abstr.*, **46**, 8857 (25 Sept. 1952).

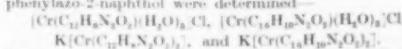
To formaldimethone (I) (2.92 g.) dissolved in 2 vols. NaOH (80 c.c.) at 15°C. was slowly added a diazonium soln. prepared from aniline (1.86 g.) in 20% HCl (10 ml.) and NaNO<sub>2</sub> (1.45 g.) in water (20 ml.). The precipitate formed after 2 hr. stirring was discarded. Acidification precipitated the red dye, bis(2-hydroxy-6-keto-4,4-dimethyl-5-phenylazocyclohexyl)methane (II) in nearly quantitative yield. I was coupled with *o*-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 2,3-xylyl,  $\alpha$ -naphthylamine,  $\beta$ -naphthylamine, *p*-BrC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, and benzidine to give dyes analogous to II. They give fast yellow-to-orange dyeings on animal fibres but are fugitive on cotton. C. O. C.

**Relation between Absorption Spectra and Chemical Constitution of Dyes. XXV—Phototropism and *cis-trans* Isomerism in Aromatic Azo Compounds.** W. R. Brode, J. H. Gould, and G. M. Wyman. *J. Amer. Chem. Soc.*, **74**, 4641-4646 (20 Sept. 1952).

During spectrophotometric studies of the photochemical *cis-trans* isomerisation of some amino- and hydroxy-azo dyes dissolved in benzene, isomerisation was so rapid that with most of the dyes reproducible spectral absorption curves could be obtained only by measuring them with the aid of a rotating shutter which allowed essentially simultaneous irradiation of the solutions during their measurement. The most probable spectral absorption curves of the *cis* forms of the dyes were calculated; those for two typical dyes are given, and the spectral absorption data for the others are tabulated. The spectra of *o*-hydroxyazobenzene and its derivatives were only slightly, or not at all, affected by irradiation, probably because of hydrogen bonding. Correlations between absorption spectra, chemical structure, and spatial configuration of the dyes are discussed. C. O. C.

**Spectrochemical Studies of Complex Dyes. V—Metallic Compounds of Dihydroxyazo Dyes.** T. Uemura and N. Hirasawa. *Bull. Tokyo Univ. Eng.*, **12**, 226-241 (1943); *Chem. Abstr.*, **46**, 7871 (10 Sept. 1952).

The ultraviolet absorption spectra of the following derivatives of *o*-dihydroxyazobenzene and *o*-hydroxyphenylazo-2-naphthol were determined—



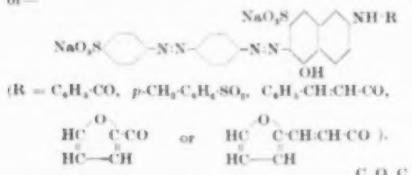
The spectrum of a stable complex consists of three bands; that of an unstable complex is one indistinct band.

C. O. C.

**Azo Dyes and their Intermediates.** X—**Synthesis of Dyes of the Benzo Fast Red Series.** T. Nishi and M. Ikenuchi. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **53**, 77-78 (1950); **XI—Synthesis of Direct Dyes in the Acyl-I Acid Series.** *Ibid.*, 118-120; *Chem. Abs.*, **46**, 8376 (10 Sept. 1952).

**X**—The synthesis and dyeing properties are described of the di-*Na* salt of 2-[*p*-(*p*-sulphophenylazo)phenylazo]-1-naphthol-3-sulphonic acid substituted in the 6 position by 2,4- and 3,5-dinitrobenzamido and 2,4- and 3,5-diaminobenzoamido radicals respectively.

**XI**—The synthesis and dyeing properties are described of—



**Malonylhydrazine Colour Formers.** R. Thiers and A. van Dornael. *Science et Ind. phot.*, **23**, 173-176 (1952); *Chem. Abs.*, **46**, 7913 (10 Sept. 1952).

**a**—and **as** **Malonylhydrazines** (I and II respectively) and **malonylmonohydrazones** (III) form yellow to red images when 0.2 g. in 10 c.c. of ethanol is added to 100 c.c. of a  $(C_6H_5)_2N-C_6H_5NH_2$  developer. The I and III are readily produced from  $CH_3(CO-NH_2)_2$  (IV) and to 2 or 1 mol., respectively, of an aldehyde or ketone in hot ethanol. The II are obtained by successively condensing IV with 1 mol. each of two different CO derivatives. High concn. of ethanol in the developer inhibits chromogenic development, whereas addition of 1 c.c. 2*N*-NaOH to 100 c.c. developer facilitates solution of the hydrazones and coupling. The I prepared from the following are tabulated: *o*-HO-C<sub>6</sub>H<sub>4</sub>CHO, yellow; *p*-peronal, yellow; C<sub>6</sub>H<sub>5</sub>CHO, yellow-orange; *o*-NaO<sub>2</sub>S-C<sub>6</sub>H<sub>4</sub>CHO, yellow; *p*-HO-C<sub>6</sub>H<sub>4</sub>CHO, yellow-orange; acetone, yellow; furfuraldehyde, yellow-orange; isatin, red. **Salicylaldehyde**, **piperal**, **malonylhydrazones** and **salicylaldehyde**, **o**-chlorobenzaldehyde, **malonylhydrazones** yellow. **For comparison** the colours produced by similar development with the following compounds are:

C<sub>6</sub>H<sub>5</sub>CH:N-NH-CO-CH<sub>2</sub>COOC<sub>2</sub>H<sub>5</sub>, yellow-orange;  
*o*-OH derivative of the preceding, yellow;  
*p*-Cl-C<sub>6</sub>H<sub>4</sub>CO-NH-NH-CO-CH<sub>2</sub>CO-NH-NH<sub>2</sub>HCl, none;  
*p*-Cl-C<sub>6</sub>H<sub>4</sub>CO-NH-NH-CO-CH<sub>2</sub>CO-NH-NH<sub>2</sub>H<sub>2</sub>O, none;  
*o*-CH<sub>3</sub>CO-CH<sub>2</sub>CO-NH-C<sub>6</sub>H<sub>5</sub>, yellow.

C. O. C.

**Heterocyclic Malonamides and Malonamates.** R. Thiers and A. van Dornael. *Bull. Soc. chim. Belg.*, **61**, 245-252 (June-July 1952).

Diethyl malonate with the 2-amino derivatives of 4-methylthiazole, 4-phenylthiazole, benzothiazole, and 5-chloropyridine at 175°C. in the absence of solvent gave the corresponding ethyl malonamates.

R<sub>1</sub>-NH-CO-CH<sub>2</sub>COOC<sub>2</sub>H<sub>5</sub> (I).

These with a different 2-amino heterocyclic base gave  $\alpha$ -malonamides R<sub>1</sub>-NH-CO-CH<sub>2</sub>CO-NH-R<sup>a</sup> (II). Both types of compounds (I and II) were formed on heating diethyl malonate with the 2-amino heterocyclic compound in the presence of sodium ethoxide and ethanol. Ethyl chloroformylsuccinate with the 2-amino derivatives of 4-methylthiazole, benzothiazole, and pyridine also gave the corresponding malonamides (I). *NN'*-Bis(3-methyl-2-benzothiazolylidene)malonamide has been obtained from diethyl malonate and 3-methyl-2-imino-2,3-dihydrobenzothiazole.

C. H. R.

**Syntheses of Heterocyclic Nitrogen Compounds.** **LVIII—Photosensitising Dyes** I. T. Takahashi and K. Satake. *J. Pharm. Soc. Japan*, **71**, 905-911 (1951); *Chem. Abs.*, **46**, 7915 (10 Sept. 1952).

**Syntheses of a wide range of trimethin dyes are given.**

C. O. C.

**Colour and Constitution. II—Aromatic Hydrocarbons.** M. J. S. Dewar. *J.C.S.*, 3532-3544 (Sept. 1952).

An extension of the treatment, applied in Part I to basic dyes (cf. *J.S.D.C.*, **66**, 653 (1950)), is described by which the first absorption maxima of aromatic hydrocarbons can be predicted with very little labour. The absorption spectra of such compounds are discussed, and the agreement between the predicted and observed wavelengths is found to be remarkably good. Agreement is bad, however, for the peropyrenes, and reasons are given for ascribing this failure to an unusually large fixation of certain double bonds in them. Data for 93 (polycyclic) hydrocarbons are included, and an appendix discusses the validity of the present use of perturbation theory.

H. H. H.

**Colour and Constitution. III—Polyphenyls, Polyenes, and Phenylpolyenes; and the Significance of Cross-conjugation.** M. J. S. Dewar. *J.C.S.*, 3544-3550 (Sept. 1952).

The procedure of Part II (above) is extended to a study of light absorption in polyphenyls, polyenes, and phenylpolyenes. Data are quoted for 30 compounds, from which the observed and calculated wavelengths ( $\lambda_0$ ) of the first absorption bands agree remarkably well. An expression is derived for  $\lambda_0$  in polyenes, in terms of the number of conjugated double bonds, which agrees much better with experiment than does the currently accepted square-root relationship. The significance of cross-conjugation is analysed, and the currently accepted rule for the light absorption of cross-conjugated alternant hydrocarbons is thereby derived.

H. H. H.

**Metallic Model of Thionine and Methylene Blue. Analysis of a Quantum Theory of Coloured Indicators.** S. Nikitin. *J. Chim. phys.*, **49**, 175-184 (1952); *Chem. Abs.*, **46**, 8519 (25 Sept. 1952).

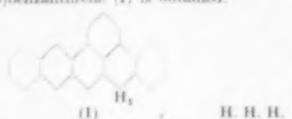
The  $\pi$ -electrons (two for each double bond) of an indicator are assumed to form a one-dimensional electron gas in a field of uniform potential energy whose length  $L$  is approx. that of the conjugated system. The potential-energy function being that of a one-dimensional potential well with rigid walls, the energy levels are given by  $E_k = k^2 \hbar^2 / 8mL^2$  ( $k = 1, 2, 3, \text{ or } 4$ ;  $\hbar = \text{Planck's constant}$ ;  $m = \text{electronic mass}$ ). Thus in the thionine molecule the seven lowest levels are occupied by the fourteen electrons which correspond to the seven conjugated double bonds. As  $L$  cannot be measured, the wavelength  $\lambda_1$  of the maximum of the first absorption band in the visible is used, so that  $E_k = k^2 \hbar^2 \lambda_1 / (2n + 1)$  ( $c = \text{velocity of light}$ ;  $n = \text{quantum number of the highest occupied level}$ ). With the help of the parameter  $\lambda_1$  the wavelengths of the other absorption bands can therefore be calculated by the formula  $\lambda = \lambda_1(2n + 1)(q + k)(q - k)$  ( $k$  and  $q = \text{quantum No. of the lower and upper levels between which the transition occurs}$ ). As the acidity of the indicator solution is increased, ions of increasingly more positive valency are formed. For every proton thus becoming attached to the ion, two electrons are borrowed from the electron gas, so that the number of occupied energy levels is reduced by one. Accordingly, certain new transitions may become possible, while certain others may become barred. However, if the wavelength of one absorption band of one of the forms of an indicator is known ( $\lambda_1$ ), the wavelengths of all the absorption bands of all forms of this indicator can be calculated. Decolorisation of indicators during pH changes is regarded as an interruption of metallic resonance. While this theory can explain why some dyes do not change their colour with pH, it cannot explain the spectrum of indicators whose absorption bands in the less acidic form are displaced toward longer wavelengths with respect to those of the more acidic form. In the cases of thionine and Methylene Blue agreement between theory and experiment is very good.

C. O. C.

**Synthesis of Naphtho[2'3':4'5]benzanthrene (4,5-9-10-Dibenzobenzanthrene (2)).** K. Holzer, E. Ziegler, and A. Zinke. *Sitzungsber. Öster. Akad. Wiss.*, **161**, 1-2, 180-184 (1952).

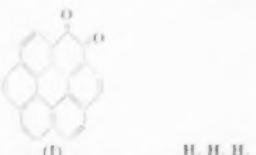
3-Chloro-2-naphthoyl chloride and phenanthrene condense with  $AlCl_3$  in CS<sub>2</sub> to give 9-(3-chloro-2-naphthoyl)phenanthrene, which, when treated with NaOH in boiling

quinoline, affords *o*-hydroxy-4,5,9,10-dihenzobenzanthrone, from which on distillation with Zn dust the naphtho(2',3',4,5)benzanthrene (I) is obtained.



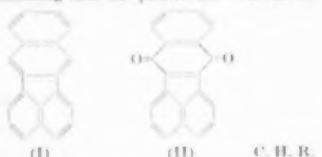
**Coronene. II—Coronene-1,2-quinone.** A. Zinke and R. Ott, *Naturforsch. Öster. Akad. Wiss.*, **16**, (3-4), 546-548 (1952).

Coronene (1 g.) in boiling nitrobenzene (30 c.c.) containing glacial acetic acid (5 c.c.), when treated for 1 hr. with a solution of sodium dichromate (4 g.) in glacial acetic acid, gives coronene 1,2-quinone (I) (1 g.); this when crystallised from nitrobenzene or sublimed at  $\sim 340^{\circ}\text{C}$ . forms long fine dark violet brown needles. It gives an orange vat, and an oxygen-free diazine with *o*-phenylene-diamine.



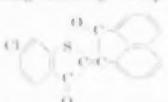
**Isolation of 8,9-Benzofluoranthene from a Coal-tar Fraction.** A. S. Harris, *Nature*, **170**, 461 (13 Sept. 1952).

This compound (I) was obtained by crystallising a neutral coal-tar fraction, b.p. 269-279 c./5 mm. Its  $\text{p}_{\text{K}_{\text{a}}}$ , 1,3,5-trinitrobenzene derivative, 2,4,7-trinitrofluorene derivative, and ultraviolet absorption spectrum were identical with those of the synthetic I. Oxidation gave a quinone, which oxidised further to naphthalic anhydride, indicating that the quinone has structure II—



**Dyes from Acenaphthenequinone. XI—6-Chloro-2,7-thionaphthacenaphthenequinone-Lindigos.** S. K. Guha and A. K. Sinha, *J. Indian Chem. Soc.*, **29**, 415-418 (June 1952).

6-Chloro-3-hydroxythiophenanthrene was condensed with acenaphthenequinone, its 3-chloro, 3-bromo, and 3-methoxy derivatives, and with phenanthraquinone to give the corresponding thiindigo compound, e.g.—



The dyes were lighter in colour than the corresponding ones obtained from 5-chloro-3-hydroxythiophenanthrene. Those from acenaphthenequinone and its halogenated compounds gave soluble purple leuco compounds which dyed cotton orange-red. The dye from the methoxy compound only partly vatted with great difficulty. The phenanthraquinone dye gave a yellow vat, which dyed cotton violet.

C. H. R.

**Syntheses of Organic Fluorescent Compounds. I—Syntheses of Iminazolone Derivatives from Acylins and Urea.** R. Oda and Z. Yoshioka, *Bull. Inst. Chem. Research, Kyoto Univ.*, **26**, 89-90 (1951).

**II—Syntheses of Iminazolone Derivatives from Aromatic Ureides and Benzoin.** *Ibid.*, **90**; *Chem. Abstr.*, **46**, 8100-8101 (10 Sept. 1952).

I—As the ureido group in the iminazolone ring is

thought to be a kind of  $-M$  substituent from the type of its resonance, the ring should, from the authors' theory (*Chem. Abstr.*, **46**, 6939 (1952)) of the fluorescence of organic compounds, be a favourable constituent for fluorescence. The yields (%) of the iminazolones made were: 4,5-diphenyl, 96; 4,5-diphenylthio, 57-8; 4,5-bis(3,4-methylenedioxyphenylthio), 27, and 4-phenyl-5-*p*-dimethylaminophenyl, 76, 0-1%. Alcoholic solutions of the diaryliminazolones did not fluoresce strongly under a quartz lamp (3650 Å.) except in the case of 4,5-diphenyliminazolone. The reasons may be that—(1) the  $+M$  effect of  $>\text{C=O}$  is greater than that of  $>\text{C-S}$ , (2) the methylenedioxy group at the 3,4-positions of the phenyl group makes the planar configuration of the molecule difficult, and (3) the effect of the  $\text{N}(\text{CH}_3)_2$  group in the *p*-*o* position of the phenyl group does not agree with the authors' theory.

II—The following yields (%) of iminazolones were obtained: 1,4,5-triphenyl, 21; 1-*a*-naphthyl-4,5-diphenyl-1,3,4,5-tetraphenyl-12,2,1-(*acetamido*-4-diphenyl)-4,5-diphenyl, 30; 1-(*acetamido*-3,3'-dimethyl-4-diphenyl)-4,5-diphenyl, 13-7, and 1-(*acetamido*-3,3'-dimethoxy-4-diphenyl)-4,5-diphenyl, 10-7, 0-1%. Solutions of these polaryliminazolones in alcohol fluoresce blue-violet to greenish blue under a quartz lamp (3650 Å.), the emission intensity decreasing as the number of substituents increases and as the aryl group becomes larger.

C. O. C.

**Observations concerning Polymorphic Crystalline Modifications of the Phthalocyanines.** F. W. Karasz and J. C. Debus, *J. Amer. Chem. Soc.*, **74**, 4716-4717 (20 Sept. 1952).

The  $\alpha$ -form of metal free phthalocyanine is produced at sublimation pressures (Barrett *et al.*, *J.C.S.*, 1719 (1936)) up to 50 mm. The  $\alpha$ -form of copper phthalocyanine is produced at pressures up to 0.1 mm.; above this pressure sublimation produces the  $\beta$ -form. An  $\alpha$ - $\beta$ -phase transition occurs for both metal-free and copper phthalocyanines above 200  $^{\circ}\text{C}$ . Barrett's condensation temperature was 400  $^{\circ}\text{C}$ , which explains why the  $\alpha$ -form of metal-free phthalocyanine was never produced by his procedure.

C. O. C.

**Infrared Absorption Spectra of Porphyrins.** C. W. Craven, K. R. Reissmann, and H. L. Chin, *Anal. Chem.*, **24**, 1214-1215 (July 1952).

The infrared absorption spectra of bilirubin, protoporphyrin, deuteroporphyrin, and coproporphyrin I and III are given, and briefly discussed. Deuteroporphyrin differs from the others in showing no marked absorption in the 5.7-8.5  $\mu$  range, this being of potential value in identifying the pigment. The log (transmittance)-concentration plot of soln. of coproporphyrin III in  $\text{C}_2\text{H}_5$ , measured at 5.72  $\mu$ , (at which all except deuteroporphyrin absorb strongly) shows a straight-line relationship.

J. W. D.

**Formation of Tyrosine Melanin. III—Use of Carbonyl-labelled Tyrosine and Dihydroxyphenylalanine in Melanin Formation.** G. R. Clemo, F. K. Duxbury, and G. A. Swan, *J.C.S.*, 3464-3468 (Sept. 1952).

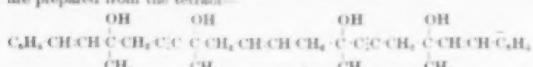
The synthesis of  $\text{mL}[\text{carboxy-}^{14}\text{C}]\text{tyrosine}$  and 3,4-dihydroxyphenyl- $\text{mL}[\text{carboxy-}^{14}\text{C}]\text{alanine}$  are described. It is found that, during melanin formation from these compounds by oxidation, nearly half of the  $\text{CO}_2$  evolved arises from carbon atoms of the amino acid mol. other than the  $\text{COOH}$  group. Moreover, 2-(3,4-dihydroxyphenyl)-ethylamine and 5,6-dihydroxyindole evolved  $\text{CO}_2$  during their aerobic conversion into melanin. It is also found that the melanin obtained from the amino acids retains some of the original  $\text{COOH}$  group.

H. H. H.

**Carotenoid Syntheses. X—Further Stereoisomeric 3,7,12,16-Tetramethyl-1:18-diphenyloctadecacarpanes. Also a Contribution to L. Pauling's Theory of Steric Hindrance in *cis*-*trans*-Isomeric Polyenes.** C. F. Garbers, C. H. Eugster, and P. Karrer, *Helv. Chim. Acta*, **35**, 1830-1864 (Oct. 1952).

The three polyenes, 3,7,12,16-tetramethyl-1:18-diphenyloctadeca-1,3,7,9(11,15,17-heptaene-5,13-diene) (I), 3,7,12,16-tetramethyl-1:18-diphenyloctadeca-1,3,7,9(11,13,14,15,17-hepteno-5-*yne*) (II), and 3,7,12,16-tetramethyl-1:18-di-

phenyltetradea-1,3:4-5:7-9:11-13:14-15:17-undecaene (III), are prepared from the tetraolein—



Partial reduction of I gives *cis*(5:6-13:14)-3:7:12:16-tetramethyl-1:18-diphenyltetradeaenone, while other *cis* forms are obtained by partial reduction of II and III. I thus possesses a *cis*-configuration at those double bonds at which, according to L. Pauling's theory (cf. *ibid.*, **32**, 2241 (1949)), it should be impossible, and the same applies to a certain extent to the other *cis* forms isolated.

H. H. H.

**Yellow Colouring Matters of Ergot.** A. Stoll, J. Renz, and A. Brack. *Helv. Chim. Acta*, **35**, 2022-2034 (Oct. 1952).

The yellow optically active compounds scopolamine acid  $\text{C}_9\text{H}_{18}\text{NO}_2$  and chrysanthemic acid  $\text{C}_{10}\text{H}_{18}\text{NO}_2$  are isolated in crystalline form from ergot, and their properties described, together with their decom. reactions with hot acetic anhydride and alkali fusion.

H. H. H.

**Potentiometric Studies of the Composition of Complex Ferrous Ferrocyanide.** A. K. Bhattacharya and R. S. Saxena. *J. Indian Chem. Soc.*, **29**, 535-538 (July 1952).

The composition of ferrous ferrocyanide, as determined potentiometrically from the  $\text{Fe}^{2+}/\text{Fe}^{3+}$  redox potential, is  $\text{K}_2\text{Fe}^{2+}[\text{Fe}^{3+}(\text{CN})_6]$ . Only the titrations in which the potassium ferrocyanide was added to the ferrous sulphate gave satisfactory results. The addition of alcohol to the titrations suppressed the adsorption of  $\text{Fe}^{2+}$  by the precipitated complex, and thus gave results nearer to the theoretical titre values.

C. H. R.

**Thermometric Studies of the Composition of Complex Ferrous Ferrocyanide.** R. S. Saxena and A. K. Bhattacharya. *J. Indian Chem. Soc.*, **29**, 632-635 (Aug. 1952).

Thermometric titrations between ferrous sulphate and potassium ferrocyanide at various concentrations suggest that  $\text{K}_2\text{Fe}^{2+}[\text{Fe}^{3+}(\text{CN})_6]$  is first formed, and this with an excess of ferrous sulphate becomes  $\text{Fe}_2[\text{Fe}(\text{CN})_6]_2$ . In the reverse titrations only  $\text{K}_2\text{Fe}[\text{Fe}(\text{CN})_6]$  is obtained. The addition of alcohol to the titrations gives results nearer to the theoretical values, owing to the decrease in adsorption of  $\text{Fe}^{2+}$  and  $\text{Fe}(\text{CN})_6^4-$  by the precipitate.

C. H. R.

**Thermometric Studies of the Composition of Turnbull's Blue Ferrous Ferricyanide.** A. K. Bhattacharya and R. S. Saxena. *J. Indian Chem. Soc.*, **29**, 529-534 (July 1952).

Thermometric titrations between ferrous sulphate and potassium ferricyanide at various concentrations suggest that  $\text{KFe}^{2+}[\text{Fe}^{3+}(\text{CN})_6]$  is first formed, and this with an excess of ferrous sulphate changes to  $\text{Fe}_2[\text{Fe}(\text{CN})_6]_2$ . In the reverse titrations, although carried out in the presence of excess ferrous sulphate, only  $\text{KFe}[\text{Fe}(\text{CN})_6]$  is formed. The slight discrepancies between the calculated and the observed titre values are explained by the tendency of the compounds formed to hydrolyse, as it was found that the addition of alcohol to the titrations gave better results.

C. H. R.

**A Study in Black.** G. J. Duffy. *Official Digest Federation Paint & Varnish Production Clubs*, (312), 28-32 (1951); *Chem. Abstr.*, **46**, 8385 (10 Sept. 1952).

Manufacture and properties of channel and furnace black are described. Optimum dispersion in a ball mill occurs with a paste containing as much carbon black as possible and with only enough paste to fill the voids between the balls. In alkyd enamels as much as 10% moisture on the black helps to stabilise consistency.

C. O. C.

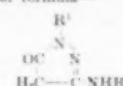
\* PATENTS

**1-Nitroanthraquinone-3-carboxylic Acid.** Basf. *BP* 678,608

1-Amino-4-nitroanthraquinone-2-carboxylic acid is diazotised by adding  $\text{NaNO}_2$  to its soln. in  $\text{H}_2\text{SO}_4$  at 20-30°C. The diazonium compound, separated by pouring into water, is reduced in ethanol with  $\text{Cu}_2\text{O}$ , the diazonium group being thereby replaced by  $\text{H}$ . Alternatively the reduction can be carried out in the  $\text{H}_2\text{SO}_4$  solution, after diazotising, by adding paraformaldehyde, and heating finally to 90°C.

R. K. F.

**Colour Couplers.** Kodak. *BP* 680,474  
Compounds of formula—



( $\text{R}^1$  = a mononuclear cyanosaryl radical;  $\text{R}^2$  = an acyl radical derived from a subst. or unsubst. aliphatic or heterocyclic monocarboxylic acid, from benzoic acid, or from an alkyl-, nitro-, halogeno-, cyano-, subst. hydroxy-, or subst. amino-benzenemono-carboxylic acid) are colour couplers for magentas of improved absorption characteristics.

*BP* 680,488

Acylamino-1-phenyl-5-pyrazolones having  $>1$  halogeno substituent on the 1-phenyl radical are excellent colour couplers for magentas.

*BP* 680,489

Pyrazolone colour couplers readily react with some hydroxyl and *gem*-diol compounds, e.g. xanthihydroxyl or alloxan hydrate, to yield monomeric and bis-pyrazolone compounds of good stability, which on coupling yield extremely stable dyes.

C. O. C.

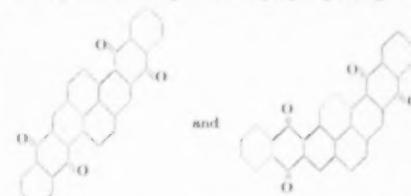
**Aromatic Colour Couplers containing Thiol and Hydroxyl Groups.** Eastman Kodak Co. *USP* 2,596,755

Compounds containing  $>1$  aromatic ring, one of which has a thiol group attached to it and another has a group which renders the compounds capable of coupling with the oxidation product of the developing agent, when used as colour couplers yield quinoneimine or azomethin dyes.

C. O. C.

**Diphthaloylpyrenes.** F. Clar. *BP* 678,172

Pyrene is condensed with  $>2$  mol. of phthalic anhydride or a nuclear-substituted derivative in a high-boiling solvent, e.g. trichlorobenzene, in presence of  $\text{AlCl}_3$ . The resulting mixture of two bis-*o*-carboxybenzoylpyrenes is separated by crystallising from dil. aq.  $\text{NaOH}$ , and the resulting isomers are cyclised in  $\text{H}_2\text{SO}_4$  to give, e.g.—

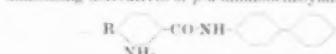


which are intermediates for vat dyes.

R. K. F.

**Monoazo Pigments.** O. F. Schulz and P. H. Fickel. *BP* 679,275

Pigments for colouring rubber, synthetic resins, cellulose nitrate lacquers, etc., or for preparing lakes, are made by diazotising derivatives of *p*-3-aminobenzoylaminodiphenyl—

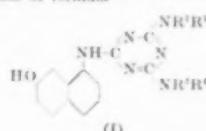


( $\text{R}$  = Alk, Alk-O, or Hal) and coupling with arylamides of 2,3-hydroxynaphthoic acid. Thus *p*-(3-amino-4-methoxybenzoyl)aminodiphenyl-2,3-hydroxynaphtho-5'-chloro-2'-4'-dimethoxyanilide, incorporated into plasticised polyvinyl chloride, gives a bluish red which does not bleed into undyed polymer.

E. S.

**Chromable Monoazo Wool Dyes.** Ciba. *BP* 679,782

Diazotised *o*-aminophenolmonosulphonic acids coupled with compounds of formula—



(I)



metal cyanide. Thus the hydrochloride of 1-amino-4- $\beta$ -methoxyethylaminoanthraquinone-2-sulphonic acid, prepared by condensing 1-amino-4-bromanthraquinone-2-sulphonic acid with  $\beta$ -methoxyethylamine, is heated for 8 hr. at 80–85°C. with aq. NaCN containing  $\text{NaHCO}_3$ .

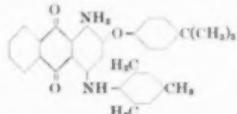
R. K. F.

**1 - Acylamino - 6 : 7 - dichloroanthraquinones—Vat Dyes.** Ciba. *BP* 679,588

Yellow vat dyes are made by acylating 1-amino-6,7-dichloroanthraquinone (nitrate and reduce 2:3-dichloroanthraquinone) with a  $p$ -halogeno(*o*-benzenesulphonyl)-benzoyl chloride. Thus 1-amino-6,7-dichloroanthraquinone is heated at 130–135°C. for 3 hr. with *p*-chlorobenzoyl chloride in nitrobenzene. R. K. F.

**1 - Amino - 4 - arylamino - 2 - phenoxyanthraquinone Sulphonic Acids—Wool Dyes.** S. *BP* 678,983

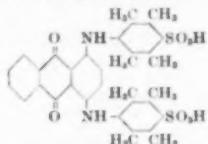
1-Amino-4-ditor-tri or tetramethylanilinoanthraquinone-2-sulphonic acid, or the corresponding 2-bromo compound, is condensed with an alkylphenol. The resulting 2-phenoxy derivatives are sulphonated to give bright violet dyes fast to light and milling. Thus the dye—



is made by heating *K p-tert*-butylphenoxide at 180°C. with 1-amino-4-(2:6:4:6-trimethylanilino)anthraquinone-2-sulphonic acid until a sample is insoluble in water. The corresponding wool dye is obtained by sulphonating with 9% oleum at 40°C. R. K. F.

**Tetramethylanilinoanthraquinones—Wool Dyes.** Allied Chemical & Dye Corp. *BP* 678,525

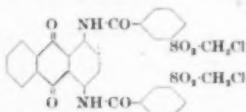
Anthraquinonoid wool dyes having good neutral-dyeing properties are made by condensing a halogenodurene (1-halogeno-2,3,5,6-tetramethylbenzene) with an *o*-amino-anthraquinone, and sulphonating. Thus the blue dye—



is prepared by heating 1,4-diaminanthraquinone, mono-bromodurene, *K* acetate, and a Cu salt in nitrobenzene at 180–190°C. for 15 hr., and finally sulphonating the product with 100%  $\text{H}_2\text{SO}_4$  at 20–25°C. R. K. F.

**Halogenomethylsulphonylbenzoylaminoanthraquinones—Vat Dyes.** ICI. *BP* 678,087

*o*-Aminonanthraquinones are condensed with mono- or di-halogenomethylsulphonylbenzoyl chlorides to give vat dyes faster to soda boiling than the corresponding unhalogenated products. Thus the dye—



is made by first boiling a mixture of *m*-chloromethylsulphonylbenzoic acid, thionyl chloride, and  $\text{ZnCl}_2$  until clear, and then heating with *o*-dichlorobenzene, 1,4-diaminanthraquinone, and pyridine at 140°C. for 3 hr. The *m*-chloromethylsulphonylbenzoic acid is prepared by condensing *Na* dichloroacetate with the disodium salt of *m*-sulphomethobenzoic acid in aqueous solution. R. K. F.

**4 : 5 - Bis - 2' - anthraquinonylamino - 1 : 8 - bis - benzoylaminoanthraquinone—Vat Dye.** Ciba. *BP* 677,381

1,8-Diamino-4,5-dinitroanthraquinone is benzoylated in nitrobenzene at 195°C., and after separating is reduced at

90–110°C. with hydrazine hydrate in nitrobenzene. The product is then condensed with 2-chloroanthraquinone at 140–150°C. in naphthalene containing *anhyd. Na acetate*,  $\text{MgO}$ , and  $\text{CuCl}_2$ . Alternatively, condensation may precede benzoylation. The vat dye so produced is olive green.

R. K. F.

**Anthraquinonecarboxyhydrazines.** DuP. *USP* 2,567,132

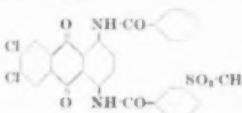
Hydrazine derivatives of general formula—



(Y =  $\text{NO}_2$  or  $\text{NH}_3$ ; R = phenylene, diphenylene, or naphthylene), useful for the preparation of the red bis-anthraquinone-oxadiazole vat dyes of *USP* 2,511,018 (*J.B.D.C.*, 57, 160 (1951)), are prepared by condensing 1-amino-1-nitroanthraquinone-2-carboxyhydrazide with the acid chloride of an aromatic dicarboxylic acid. Thus 1-nitroanthraquinone-2-carboxyhydrazide and terephthaloyl chloride are heated in nitrobenzene at 165–170°C. for 2 hr. R. K. F.

**4 - Alkylsulphonylbenzoylamino - 1 - benzoylamino - 6 : 7 - dichloroanthraquinones—Vat Dyes.** Ciba. *BP* 680,378

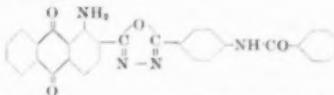
Red to violet acylaminoanthraquinone vat dyes are made by acylating 4-amino-1-benzoylamino-6,7-dichloroanthraquinone with a *m*(or *p*)-alkylsulphonylbenzoyl chloride. Thus the dye—



is prepared by first heating *m*-methylsulphonylbenzoic acid in nitrobenzene with  $\text{SOCl}_2$  and a little pyridine at 110–115°C. for 1–5 hr. 4-Amino-1-benzoylamino-6,7-dichloroanthraquinone (obtained from 2,3-dichloroanthraquinone by nitrating, reducing, renitrating after protecting the  $\text{NH}_2$  group, benzoylating, and reducing) is then added, and heating is continued at 125–130°C. for a further 2 hr. R. K. F.

**Anthraquinonyl - 1 : 3 : 4 - oxadiazole Derivatives—Vat Dyes.** I.B.P. *BP* 680,364

Vat dyes and intermediates for them, containing one or more 1:3:4-oxadiazole rings, each linked directly to one anthraquinone residue, are made by first condensing an anthraquinonecarboxylic chloride with the monoacylhydrazide of a non-volatile carboxylic acid and then cyclising the resulting *NN'*-diacylhydrazine. Thus the scarlet vat dye—



is made by first refluxing *p*-nitrobenzoylhydrazide with 1-aminoanthraquinone-2-carboxylic acid in nitrobenzene. The resulting diacylhydrazine is heated at 50°C. in chlorosulphonic acid to give the corresponding oxadiazole. The nitro group is finally reduced and acylated with benzoyl chloride. R. K. F.

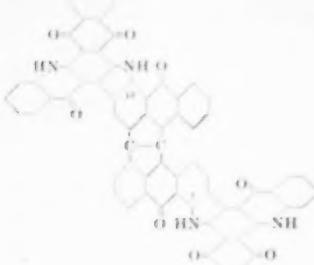
**Phthalocyanine Dyes.** Basf. *BP* 679,808

Metal or metal-free phthalocyanines are heated with polycarboxylic acid anhydrides in presence of a Friedel-Crafts condensing agent, e.g.  $\text{AlCl}_3$ , to give vat, substantive, or wool dyes depending on the quantities and conditions used. Thus cobalt phthalocyanine and succinic anhydride are heated at 150°C. for 8 hr. in a melt of  $\text{AlCl}_3$  and  $\text{NaCl}$ . A blue vat dye separates on pouring into water and acidifying. R. K. F.

**Acedianthrone Derivatives—Vat Dyes.** ICI.

BP 678,522

An olive green vat dye with good printing properties, having the formula—



is prepared by treating the condensation product of 7,15-dichloroacanthrone and 3-amino-1,2-phenylhydrazine described in *BP* 672,908 (J.S.D.C., **68**, 320 (Aug. 1952)) with an acid condensing agent, e.g.  $\text{AlCl}_3$ , in nitrobenzene at 110–115°C. The aminophthalimidone is made by the method described in *BP* 587,006 (J.S.D.C., **64**, 199 (1948)).

BP 678,523

7,15-Dichloroacanthrone, made from 1-chloro-9-anthrone and glyoxal as described in *BP* 551,622 (J.S.D.C., **59**, 155 (1943)) is condensed with 1 or 2 mol. of mono-aminocanthrone by heating at 205–210°C. for 24 hr. in nitrobenzene containing  $\text{NaClO}_2$  and a Cu salt, to produce brown vat dyes of good printing properties. R. K. F.

**Copper Phthalocyanine.** Standard Ultramarine Co. USP 2,568,569

*o*-Dihalogenobenzenes are heated with cuprous cyanide or cyanide mixtures containing cuprous cyanide to produce copper phthalocyanine. The reaction may be carried out by heating a suspension of the cyanide in the *o*-dihalogenobenzene under pressure, or the vapoured halogen compound may be passed over the cyanide supported on copper gauze, or through a fluidised bed consisting of the cyanide precipitated on finely divided inert material, e.g. diatomaceous earth. Thus aqueous potassium cuprous cyanide is slowly added to boiling *o*-dibromobenzene, the water distilled off, and refluxing continued for 4 hr. The copper phthalocyanine, which separates on cooling, is freed from excess Cu salts by treating with  $\text{H}_2\text{SO}_4$ . USP 2,568,570

Copper and other metal phthalocyanines are prepared by heating the corresponding metal phthalate with urea and a catalyst, e.g. ammonium molybdate. Thus copper phthalate, prepared by evaporating mixed solutions of phthalic acid and cupric acetate, is extracted by the resulting solid with acetone and water, and leached with urea and ammonium molybdate, and leached subsequently with HCl and KOH. R. K. F.

**Zinc Phthalocyanine Pigments.** Ciba. BP 679,773

Crude zinc phthalocyanine is converted into useful blue pigments by dry grinding with a grinding aid, e.g.  $\text{NaCl}$  or  $\text{Na}_2\text{Fe}(\text{CN})_6$ , which can be later washed out. No conversion of  $\beta$ - to  $\alpha$ -form occurs during grinding, but this takes place during the washing unless non-polar solvents are used, or during the subsequent drying at about 100°C. Pigments containing up to 100%  $\beta$  form, having a greener and purer hue than those containing mostly the  $\alpha$  form, are obtained by first wetting the ground mixture with non-ionicogenic solvents, e.g. ethanol, or alternatively by heating the final dry product to 150–200°C. R. K. F.

**Phthalocyanine Vat Dyes.** Basf. BP 678,195

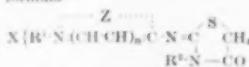
Blue dyes, some of which dye cotton directly and others from a hydroxysulphite vat, are made by heating phthalocyanines with lactones in the presence of Friedel-Crafts condensing agents. Thus cobalt phthalocyanine, anhyd.  $\text{AlCl}_3$ ,  $\text{NaCl}$ , and  $\gamma$ -butyrolactone are heated together for

## VI—FIBRES; YARNS; FABRICS

several hours at 150°C. The dye is separated by pouring into ice and water. R. K. F.

**Azamethinthiazolone Cyanine Dyes.** General Aniline. BP 680,399

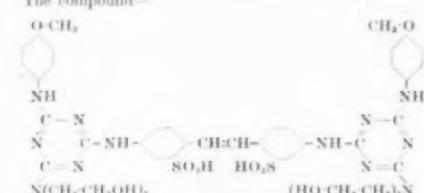
Dyes of formula—



( $\text{R}^1$  and  $\text{R}^2$  = subst. or unsubst. Alk, alkyl, Ar, or aralkyl;  $n = 0$  or 1;  $\text{X} =$  an acid radical;  $\text{Z} =$  atoms to complete a heterocyclic nucleus) are photographic sensitizers and valuable intermediates for tri- and tetra-nuclear dyes. C. O. C.

**Fluorescent Whitening Agent.** General Aniline. USP 2,595,030

The compound—



is useful for application to cellulosic materials, cellulose acetate, etc. It is faster to chlorine than known related compounds. C. O. C.

**Carbon Black.** Phillips Petroleum Co. BP 679,818–20

Production of a furnace black which is harder than channel black is described. C. O. C.

**Pearl Essence Survey** (V below).

Silicon-containing Azo Dyes. III—Dyeing Properties, Absorption Spectra, and pH Range (VIII p. 532). Sensitivity to Lime of Anionic Leather Dyes (VIII p. 534). Sensitivity to Lime of Anionic Dyes (VIII p. 534).

## V—PAINTS; ENAMELS; INKS

**Pearl Essence Survey.** A. J. Krajcman. Paint, **22**, 371–375, 387 (Oct. 1952).

A survey of the history, chemistry, and manufacture of pearl essence, essence d'orient, or fish silver, which consists of a suspension of guanine crystals (obtained from fish scales) in a clear lacquer. It is used where pearl or mother of pearl effects are required or for finishes which have to be particularly lustrous; e.g. coloured pearl essence is used to impart brass, bronze, or other finishes to simulate metal. 30 references. C. O. C.

## PATENTS

**Pelleted Pigments for Colouring Paints.** New England Paint Works. BP 680,239

Pigment is mixed with linseed oil to form a coherent pellet, which is then given a coating that is soluble in linseed oil. This enables white paint to be kept in tins, so that it can be coloured at any desired time by stirring suitable pellets into a tin of paint. C. O. C.

**Heat-set Inks containing Polyvinyl Terpene Ethers.** Hercules Powder Co. USP 2,505,983

Heat-set printing inks comprising a colouring matter dispersed in a resinous polymer of a vinyl ether of a terpene alcohol dissolved in petroleum, or other solvent which is non-volatile at 70°F. but which has high vapour pressure at 200–240°F., yield glossy, flexible, non-scuffing prints of excellent adhesion. C. O. C.

## A Study in Black. IV p. 525.

## VI—FIBRES; YARNS; FABRICS

**Photolysis and Fine Structure of Textile Fibres.** A. Sippel. *Kolloid-Z.*, **127**, 79–82 (July 1952).

A mathematical derivation is given which shows that the proportion of light-sensitive covalent bonds between the basis molecules enables the ratio of the length to the thickness of the crystallites to be deduced. From the data for

several types of fibres it is conjectured that they are thin rather than thick—a concept resembling that of Hosemann (*ibid.*, **117**, 14 (1950); **119**, 129 (1950)).

L. P.

**Method for Studying the Effect of Humidity on the Cross-sectional Swelling of some Common Fibres.** F. F. Morehead. *Text. Research J.*, **22**, 535-539 (Aug. 1952).

A cross-sectional method for studying the effect of humidity on the swelling behaviour of various fibres is detailed. A great increase in swelling between 97% R.H. and wetting was observed in the case of the rayons and cotton. A. B.

**Action of Cyanuric Chloride on Cotton Cellulose.** J. Warren, J. D. Reid, and C. Hamalainen. *Text. Research J.*, **22**, 584-590 (Sept. 1952).

The reaction of cyanuric chloride with alkali-treated cotton cellulose has been examined to determine the effect of reaction conditions on degree of substitution and on retention of chlorine for further modification. The highest substitution obtained was 1 triazine ring per 1-9 anhydroglucosidic residues, and the highest chlorine content was 1 Cl per 3-4 anhydroglucosidic residues. The chlorine was stable to several months' storage and to heating at 161°C., and was capable of reacting with ethylenediamine and hexamethylenediamine. A. B.

**Heterogeneous Hydrolysis of Highly Methylated Cotton Cellulose.** R. E. Reeves, B. J. Barrett, and L. W. Mazzoni. *J. Amer. Chem. Soc.*, **74**, 4491-4494 (20 Sept. 1952).

Heterogeneous hydrolysis of highly methylated cotton cellulose causes a sharp decrease in viscosity (in chloroform soln.), which falls to a minimum and then, upon prolonged hydrolysis, begins to increase. Different minimum viscosities for different batches of methyl cellulose seem to be caused by different crystallite dimensions produced during methylation, and this is supported by X-ray diffraction patterns of the methyl celluloses. The initial viscosity decreases are probably due to chain scission; the later increases are regarded as due to more rapid destruction of the material of lowest viscosity. C. O. C.

**Decomposition of Viscose—Dissociation Constant of Cellulose Xanthic Acid.** E. Treiber, H. Karen, W. Felbinger, and W. Lang. *Sitzungsber. Öster. Akad. Wiss.*, **161**, (3-4), 239-270 (1952).

Cellulose xanthic acid is formed during the decomposition and regeneration processes of viscose, and this has been established for freshly spun fibres. The difficulties attendant upon the experimental determination of its dissociation constant are discussed, and from several sets of independent measurements it would appear that it lies within the limits 2.1-5.5  $\times 10^{-5}$ , i.e. the acid is somewhat stronger than acetic acid. Included are the X-ray photograph of precipitated sodium cellulose xanthate and 18 references. H. H. H.

**Sorption of Water by Viscose Rayon at Low Humidities.** J. B. Taylor. *J. Textile Inst.*, **43**, T 489-T 515 (Sept. 1952).

Apparatus is devised for accurately determining low moisture sorption, and sorption data are presented for regains of  $< 2.5\%$ , at 25-65°C. for commercial viscose rayon. The adsorption isotherm is reproducible provided that prior drying does not take place at too high a temperature. Hysteresis sorption-desorption effects are found in the region up to 1-2% regain, at which values capillary mechanisms are inadmissible and swelling effects negligible; these results are discussed in the light of current hysteresis theories. The low-humidity adsorption isotherms are approximately described by the Freundlich equation. The isosteric heat of adsorption is calculated. J. W. B.

**Occurrence of Small-angle X-Ray Diffraction Maxima in some Artificial Cellulose Fibres.** D. Heikens, P. H. Hermans, and A. Weidinger. *Nature*, **170**, 369-370 (30 Aug. 1952).

Maxima obtained with Fortisan (Celanese Corp.) and Fibre G (DuP), both in the water-swollen state, correspond to Bragg spacings of 84 and 77 Å, respectively. These are interpreted as evidence of lateral periodicity in the length and arrangement of crystalline and non-crystalline regions in the fibres. Though known in highly oriented

polyamides, Terylene, and polythene, this has not previously been observed in well oriented cellulose fibres.

J. W. D.

**Nature of the Hemicelluloses of Jute Fibre**—L. P. B. Sarkar, A. K. Mazumdar, and K. B. Pal. *Text. Research J.*, **22**, 529-534 (Aug. 1952).

A polyuronide hemicellulose, constituting 75% of the hemicelluloses of jute, is isolated, and is shown to be probably 1 mol. of methylglucuronic acid linked with 6 mol. of anhydroxylose. A methyluronic acid-xylene complex is the main acidic constituent of the fibre, and is apparently responsible for the natural affinity of the fibre for basic dyes. A. B.

**Recent Researches on Wool.** H. Zahn. *Textil. Rund.*, **7**, 305-319 (July) and 398 (Aug. 1952).

Studies carried out at Heidelberg University concern the morphological structure of wool and human hair, and the chemistry of cross-linkage reactions. Progress in chemically stabilised wool may lead to a product suitable for dyeing in alkaline bath at high temperatures. 37 references. G. L.

**Moisture Relations of Textile Fibres at High Humidities.** D. K. Ashpole. *Proc. Roy. Soc. A* **212**, 112 (8th April 1952).

Hygroscopicity of textile fibres and the phenomenon of hysteresis are discussed, and it is considered that in previous experiments error has arisen through the non-saturation of supposedly saturated atmospheres. A technique for overcoming this difficulty is described, and measurements are made of the moisture relations at very high humidities of viscose rayon, cotton, isoelectric and non-isoelectric wool, Cellophane, and acid-formaldehyde-treated viscose rayon. Saturation regains much higher than those previously found are obtained, and are in close agreement with inhibition values obtained by a standard centrifuging procedure. The hysteresis loop closes at saturation. There is no true equilibrium in a saturated atmosphere; water absorption steadily increases owing either to hygroscopic salts, which tend to form an infinitely dilute solution, or to flooding of the capillary structure. J. W. B.

**Saturation Moisture Absorption of Wool.** G. King and F. L. Warburton. *J. Textile Inst.*, **43**, T 516-T 517 (Sept. 1952).

Data presented by Ashpole (*Proc. Roy. Soc. A* **212**, 112 (1952)), showing that the maximum regain of wool is 60%, are considered to be invalidated by the fact that the maximum radial swelling of a wool fibre in water is only 16.4%, which corresponds to a regain of 33%. J. W. B.

**Supercontraction of Keratin.** E. H. Mercer. *Text. Research J.*, **22**, 476-479 (July 1952).

The nature of the supercontraction of keratin fibres by about 30% of their original length is investigated. On examination in the electron microscope, fragments of supercontracted wool appear to be twisted, and it is suggested that such twisting rather than molecular folding is directly related to the length changes of the whole fibre. Little similarity between contracted muscle and supercontracted keratin is found. A. B.

**Combination of Wool with Acids.** B. Olofsson. *J. S.D.C.*, **68**, 506-510 (Dec. 1952).

**Properties of Apparel Wools. II—Modification of Fibre Surface during Worsted Processing.** D. F. O'Reilly, J. C. Whitwell, R. O. Steele, and J. H. Wakelin. *Text. Research J.*, **22**, 441-447 (July 1952).

Samples of wool taken at each stage of their manufacture into a worsted fabric were treated with Pauli reagent and examined microscopically, and the rate of exhaustion of an acid dye was also measured. An increasing amount of surface modification was observed during processing, together with an increase in staining with Pauli reagent and in rate of uptake of Anthraquinone Blue SKY. All these changes were reversed by steaming. It is concluded that the dye susceptibility of processed wool is related to the amount of scale disturbance. A. B.

**Influence of Stress on the Swelling of Animal Fibres.** O. Raja and J. B. Speakman. *J. Textile Inst.*, **43**, T 517-T 518 (Sept. 1952).

The diameters of tensioned and non-tensioned wool fibres are measured at 67% R.H. and in water. Results contrast with those of White and Stam (*Text. Research J.*, **19**, 136 (1949)), and show that within this humidity range

there is little change in diameter with rise of tension within the Hooke's law region of the load-extension curve. It is considered that White and Stam's observations may have been invalidated by failure to take into account the elliptical cross-section of their fibres. J. W. B.

**Swelling of Human Hair in Water and Water Vapour.**  
P. B. Stam, R. F. Kratz, and H. J. White. *Text. Research J.*, **22**, 448-465 (July 1952).

The swelling of fresh and strained hair fibres in water and water vapour is examined, the strained fibres being at either constant load or constant elongation. The swelling isotherms of the free fibres are found to follow the volume changes from density measurements, except at very high R.H. An attempt is made to develop a statistical-mechanical model to explain the equilibrium absorption and swelling behaviour of a hair fibre. A. H.

**X-Ray and Ultraviolet Spectrographic Investigations of Fibrous and Globular Modifications of Silk Fibroin.** O. Kratky and E. Schausenstein. *Discussions Faraday Soc.*, **(11)**, 171-178 (1951).

Silk I, prepared by allowing fresh silk gel from mature worms to dry in air, shows an X-ray pattern similar to that of *Gaein*. If it is swollen in water, NaOH, or H<sub>2</sub>CO<sub>3</sub>, stretched, and dried in the extended state, a modification is produced which the authors term *Silk II*. This latter may also be produced directly from the original gel by partial coagulation with 2% CH<sub>3</sub>COOH followed by combined rolling and stretching. Comparison of the ultraviolet absorption spectra of Silk I and II indicates that the transformation to Silk II is accompanied by emulsion of peptide groups (absorb. C(OH)<sub>2</sub>N-) and the formation of hydrogen bonds (absent from Silk I and from the original gel). The presence of many more large groups of hydrogen-bridged peptide chains is also confirmed when Silk II is examined between crossed nicols. Brief treatment with hot alkali introduces new small-angle diffraction maxima into the X-ray pattern of Silk II. That corresponding to a spacing of 45 Å is attributed to a very impurity (~1%), which may be made to crystallise under certain conditions of regularity of the intermolecular fissures. In some highly oriented silks the 45-Å. max. is replaced by one at 36 Å, this appearing without the need for an alkali treatment; the difference is attributed to soap formation. Hot alkali treatment causes complete dissociation of the phenolic groups in the tyrosine residues of freshly prepared fibroin films, this being accompanied by the appearance of a weak meridional reflection of period 21 Å. when the dissociation is considerable. Cold alkali has little or no effect, since the hydrogen-bonded lattice is not easily penetrated without the aid of heat. Aged silks show considerable (and even complete) dissociation with cold alkali, indicating that the hydrogen bonds have become loosened. Changes in the diffuse small-angle scattering in the X-ray patterns of fresh and aged silk fibres indicate that silk, like other fibrous proteins, has a large range structural plan in which tyrosine plays a decisive part, but in which glycine, alanine, and rarer amino acids also have a place. A soln. of silk in cupro-ethylene diamine (J.R.D.C., **64**, 121 (1948)) gives, on drying, a film which shows an X-ray pattern similar to that of Silk II, but of poorer lattice order. When this is swollen, the pattern resembles that of the original gel; a Silk II may be prepared from it which differs from that made from fresh gel in such a way that the damage resulting from the "renaturing" (dissolution, drying, and re-swelling) process resembles that due to the normal ageing of silk. (Cf. J.S.D.C., **68**, 410 (Oct. 1952)). J. W. D.

**Chromatographic Studies of the Silk Fibre.**  
I—Amino Acid Composition of Two Sericin Fractions. II—Fractionation of a Fibroin Hydrolysate. F. Bryant. *Text. Research J.*, **22**, 523-525, 525-528 (Aug. 1952).

I—Sericin isolated from raw silk is shown to be readily separable into water-soluble and water-insoluble fractions. When analysed for amino acid content by paper partition chromatography the two fractions showed distinct differences.

II—An acid hydrolysate of silk fibroin was adsorbed on a column of a cation-exchange resin, and ammonia used as displacement developer, but very little separation was achieved. A. H.

**Static Electricity in Textiles.** S. M. Edelstein. *Amer. Dyestuff Rep.*, **41**, P. 518-P. 524 (18 Aug. 1952).

New synthetic fibres which have low moisture absorption cause more static trouble than the natural fibres. These troubles may occur at all stages of processing from fibre blending to wet finishing, and take many forms, including fibre-fibre repellency, leading to difficulties in carding, spinning, and weaving. Finished fabrics fold and crease badly, and attract dirt and lint. Methods of preventing these difficulties are discussed, and include the use of radioactive materials, humidity control, earthing devices, and the use of applied chemical antistatic agents. A. H.

**Microscopy of Nylon.** R. Lassé. *Textil-Rund.*, **7**, 353-359 (Aug. 1952).

ZnCl<sub>2</sub>-KI reagent produces nodules and contractions along nylon fibres similar in appearance to the initial effect of cuprammonium on cotton. It is likely that the outer skin of the fibre, consisting of densely packed parallel chains, is affected by swelling and staining. Continuation of treatment increases the thickness of the swollen layer, and crossing fibres fuse at their points of contact. Finally, hydrolysis of nylon occurs, leaving oval residues, whilst any admixed wool remains unchanged. Dry fibres may take up to two days to reach the final stage, but the period can be reduced to 25 min. by preliminary wetting out in 80% alcohol on the microscope slide. The action of chloral hydrate and the applications of polarised light are also briefly described. 17 micrographs. G. L.

**Electrolytic Interaction of Nylon with Aqueous Sodium Hydroxide.** F. T. Wall and T. J. Swoboda. *J. Physical Chem.*, **56**, 50-56 (1952); *Chem. Abs.*, **46**, 7847 (10 Sept. 1952).

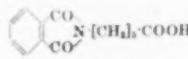
Gilbert and Rideal's theory of the interaction of charge-bearing fibres with acids and bases is generalised to cover the six possibilities of titration with either acid or alkali of nylons having  $A > B$ ,  $A = B$ ,  $A < B$  respectively ( $A$  = total COOH groups;  $B$  = total NH<sub>2</sub> groups). One case, titration with alkali when  $A > B$ , was investigated by equilibrating undrawn fibres (nylon 66), containing  $82 \times 10^{-4}$  mol. of A/g. and  $42 \times 10^{-4}$  mol. of B/g., with varying strengths of aq. NaOH so that absorption varied from nil to saturation. Good agreement is observed between theory and experiment, and the results are used to calculate equilibrium constants for ion absorption and neutralisation processes. C. O. C.

**Perlon.** J. Wrieth. *Textil-Rund.*, **7**, 323-326 (July 1952).

The history, method of production, and properties of the fibre are outlined. It is claimed that Perlon L can be manufactured as a continuous process with comparatively simple equipment. G. L.

**Structure of the Reaction Product of  $\epsilon$ -Caprolactam and Phthalic Anhydride.** G. M. van der Want. *Rec. Trav. chim.*, **71**, 1012-1016 (Sept.-Oct. 1952).

The reaction product of  $\epsilon$ -caprolactam and phthalic anhydride is found to be identical with that of  $\epsilon$ -aminocaproic acid and phthalic anhydride, so that its structure is that of  $\epsilon$ -phthalimidocaproic acid (6-phthalimidohexanoic acid)—



H. H. H.

PATENTS

**Cellulose Xanthate Fibres.** American Viscose Corpn.

USP 2,594,496

Pure cellulose xanthate fibres are produced by extruding viscose into an aqueous bath containing acetic acid and sodium sulphate and/or sodium acetate. They may be stretched as they are withdrawn from the bath. Such fibres may be treated with cross-linking or other agents, or may be converted into regenerated cellulose fibres having the characteristic properties of cotton, viz. low extensibility, especially when wet, and good to superior tenacity. C. O. C.

**Lubrication of Yarn in Packages.** Celanese Corp. of America. BP 680,459

Textile material is lubricated by passing through it water containing an organic water-in-oil lubricant emulsified by means of an emulsifying agent, the proportions of these being such that the emulsion is so unstable

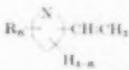
that lubricant is abstracted from the aqueous phase and deposited on the material. In one example a composition is prepared containing a mineral lubricating oil (Saybord universal viscosity 50 at 100°F.) (55 parts by wt.), Na or K oleate (12.5), and diethylene glycol monostearate (2.5). This is mixed with demineralised water, of zero hardness, to a final concn. of 300 p.p.m. This solution is used as the final wash water in the purification of yarn produced by the viscose process, and is pumped through the bobbins for 4 hr. at the rate of 9 lb. lb. yarn hr., after which the water is discarded. The yarn packages have improved rewinding properties. J. W. B.

**Dimensionally Stable Wool Acrylonitrile Polymer Yarns.** DuP. USP 2,595,977

Yarns made of wool blended with as little as 15% of a fibre made from a polymer containing at least 85% of acrylonitrile are dimensionally stable to normal washing. C. O. C.

**Acrylonitrile Copolymers readily dyed with Acid Dyes.** American Viscose Corp. BP 680,359

Ternary copolymers of acrylonitrile, a vinylpyridine of formula—



(R =  $\text{CH}_3$  or  $\text{C}_2\text{H}_5$ ; n = 0-4), and styrene, vinyl acetate, or an acrylic ester are readily formed into oriented fibres, which yield deep fast dyings with acid dyes. C. O. C.

**Dyeable Acrylonitrile Copolymers.** Chemstrand Corp. USP 2,595,575

Copolymers of acrylonitrile and 1-allyloxy-3-chlorot or bromo-2-propanol, 1-methylallyloxy-3-chloro(or bromo)-2-propanol, or 1-chlorodiallyloxy-3-chloro-2-propanol are readily formed into fibres, which when treated with a primary or secondary amine or ammonia can be dyed with acid dyes, the affinity for the dye being still further increased if they are given a further treatment with an agent which converts amino groups into quaternary ammonium salts. U.S.P. 2,595,848

The fibre-forming copolymers of acrylonitrile and the vinyl, allyl, and methyl esters of a halogenocarboxylic acids are given affinity for acid dyes by treatment with an alkali-metal derivative of a nitrogenous heterocyclic thiol, e.g. the sodium salt of mercaptobenzothiazole. C. O. C.

**Linear Polyureas.** Nederlandse Organisatie voor Toegepast-natuurwetenschappelijk Onderzoek ten Behoeve van Nijverheid, Handel en Verkeer. BP 679,671

The chain length of the polyureas obtained by treating an aliphatic diamine with carbonyl sulphide is controlled by addition of small quantities of primary or secondary amines. W. G. C.

**Strengthening Artificial Insolubilised Protein Filaments.** ICI. BP 657,686

The strength of regenerated protein filaments which have been treated with reagents capable of forming *in situ* nascent monomeric thioformaldehyde, e.g. according to BP 533,952 (J.S.D.C., 57, 277 (1941)), is improved if they are treated under tension with aq. mercuric chloride. C. O. C.

**Acrylonitrile-Protein Derivatives (III p. 521).** Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Macromolecular Poly-unsaturated Compounds (VIII, p. 534).

**Molecular State of Water bound to the Peptide Link (XII p. 537).**

**Depilatory Activity of Sodium Sulphide and Related Compounds (XII p. 538).**

**Dehairing Skins (XII p. 538).**

**VII—DESIZING; SCOURING; CARBONISING; BLEACHING**

**Emulsions and Films.** A. S. C. Lawrence. *Nature*, 170, 232-234 (9 Aug. 1952).

Summaries are given of papers presented at a symposium held by the British Society of Rheology at the University of Sheffield in March 1952. W. K. R.

**The Micelle and Swollen Micelle on Soap Micelles.**

W. Philippoff. *Dimensiones Faraday Soc.*, (11), 96-107 (1951).

The concn. dependence of the osmotic activity and of the specific conductivity of ten detergents of various types has been calc., and in each case is represented by two intersecting straight lines. From the ratio of their slopes, the micelle size and charge can be evaluated. The calc. results agree well with those obtained by other methods. These data are embodied in a general discussion of X-ray and other evidence available to date, designed to give a quantitative correlat on and general picture of "micellar solutions". The micelle is shown to be essentially a double layer of 50-100 molecules, oriented to give an X-ray pattern; they are partly ionised and hydrated. Complex or ramified molecules form smaller micelles than straight chain compounds with the same ionogenic group.

J. W. B.

**Viscometric Study of the Micelles of Sodium Dodecyl Sulphate in Dilute Solutions.** L. M. Kushner, B. C. Duncan, and J. I. Hoffman. *Ind. Stand. J. Res.*, 49, 85-90 (Aug. 1952) — *Research Paper* 2346.

Sodium dodecyl sulphate is synthesised, and concentrations up to 0.8% by wt. in distilled water and in 0.01-0.12 M NaCl, are used for viscosity measurements, using a modified Ostwald viscometer. Surface tension and density measurements are also made, and a method is presented of determining monomer saturation concentration, i.e. that point at which micelles start to form, by the light absorption at 630 m $\mu$  of Toluidine Blue in presence of the detergent. By introducing the concept of this saturation concentration it is possible to determine the intrinsic viscosity of the micelles at each NaCl concn. The dependence of intrinsic viscosity on salt concn. is discussed in terms of electroviscous and hydration effects; results suggest that micelles of this particular detergent are spherical. J. W. B.

**Influence of Detergents on the Solubility of Hydrocarbons.** H. B. Klevens. *Kolloid Z.*, 128, 61-67 (Sept. 1952).

Solutions of the cationic detergent dodecylbenzylchloride are able to dissolve 8-9 times as much ethylbenzene or anthracene as similar solutions of the anionic agent potassium dodecyl carbonate. The difference is attributed to the higher state of disorder in the anionic micelle. L. P.

**Electrophoresis of Pigment Particles in Detergent Solutions.** W. Kling and H. Lange. *Kolloid-Z.*, 127, 19-27 (June 1952).

The electrostatic charge on the particles of dirt is thought to be the major factor in washing textile materials. Its influence is investigated by measuring the electrophoretic mobility of particles of soot, coal dust, and iron oxide in solutions of detergents of different types and varied concentration. Electrophoresis shows that the negative  $\zeta$  potential of the particles increases with increasing concn. of anionic detergents such as alkaline dodecyl sulphate, laurate, or alkylbenzenesulphonate. A non-ionic detergent of the polyethylene oxide type has little effect, but in presence of salts the potential steadily falls. Neutral salts (NaCl, K<sub>2</sub>SO<sub>4</sub>) alone have no effect, although the mobility is slightly increased by alkaline salts (sodium polyphosphates, Na<sub>2</sub>O<sub>3</sub>, NaOH) and they also set a limit to the effect detergents can have. The cationic detergent, dodecylpyridinium chloride, lowers the potential until, even in minute concentration, it reverses the sign of the charge. Sodium carboxymethyl cellulose does not appreciably affect the mobility, although, if the increase in viscosity is allowed for, the potential seems to increase. On the other hand, methyl cellulose produces a sharp fall in mobility, especially in the presence of electrolytes. The phenomena agree with the known detergent action of these agents. L. P.

**Adsorption in the Detergent Process.** A. L. Meader and B. A. Fries. *Ind. Eng. Chem.*, 44, 1636-1648 (July 1952).

A radioactive tracer technique was used to study the adsorption of sodium alkylsulphonate, labelled with <sup>35</sup>S, and sodium palmitate, labelled with <sup>14</sup>C, by cotton and wool, from solution in both distilled and hard water. With each compound, increasing temperature increased the rate but decreased the magnitude of adsorption. Addition of

salts increased the adsorption of sulphonate. Desorption of the sulphonate was more complete in distilled than in hard water, but desorption of the soap was greater in hard water. Wool adsorbed more of each compound than cotton, and also held them more tenaciously on rinsing. No simple relationship was found between adsorption and detergency. W. K. R.

**Detergency Tests on Artificially Soiled Wool, using Various Synthetic Detergents in conjunction with Polyphosphates.** H. Stupel. *Textil Praxis*, 7, 723-725 (Sept. 1952).

The efficiency of various synthetic detergents (one each of six different chemical types) at 40° C. and at pH ~ 7 (no further addition) and 9 (attained by adding NaOH, tetrasodium pyrophosphate, or sodium tripolyphosphate) is assessed by means of washing tests on artificially soiled wool, using the apparatus and method of assessment already described (see J.S.D.C., 68, 272 (July 1952)). The results are compared with those already obtained for synthetic detergents on cotton (*Soap, Perfumery, and Cosmetics*, 24, 548 (1951)). The variation in detergent properties from one agent to another and the improvements effected by increase in pH and by additions of phosphates are less than for pyrophosphate, but is used at higher concn. A small addition of lauryl alcohol (0.5 g. litre) to the detergent soln. (1.5 g. litre) generally results in increased efficiency. No effect on handle is produced at this concn.; at higher concn. a soft handle is imparted, but detergency efficiency falls to about the initial value. The behaviour of the non-ionicogenic detergent is exceptional: the detergency effect is greatly reduced by a small addition of lauryl alcohol, but, when excessive amounts are used, the detergency efficiency rises again to above the initial value. A. E. S.

**Relationship between the Greying of Textiles and the Frictional Electricity generated in Organic Solvents.** G. E. F. Brewer. *J. Amer. Oil Chem. Soc.*, 29, 218-219 (1952). *Chem. Abstr.*, 46, 7334 (10 Aug. 1952).

This frictional phenomenon is important in dry cleaning in connection with the fire risk as well as the so-called tint transfer and insufficient soil removal. The frictional electrets in the solvent systems were measured with an electronic amplifier by the use of metal cells in which relative motion was produced by a rotating shaft covered by a textile sleeve. The tests showed voltage readings (~ 30%) as follows: commercial dry-cleaning fluids 0.02-0.70; Stoddard solvent (I) 0.07,  $\text{CHCl}_3\text{CCl}_4$  (II) 0.28, 40 : 60 mixture of I and II 1.50. With mixtures of I and butanol maximum frictional electricity was 78% of I. Tests carried out using colloidal graphite stearic acid suspensions in the two component systems showed that greying increased and decreased with the observed frictional electretics. C. O. C.

**Attack of Sodium Chlorite on Cellulose in Acid Medium.** F. Kocher. *Bull. Inst. textile France*, (31), 19-90 (Feb.); (32), 19-103 (April 1952).

Literature dealing with textile applications of chlorites and the structure of oxychlorose is surveyed. The principal oxidant of cellulose is the chlorite ion and not  $\text{ClO}_4^-$ . The cellulose is attacked only at high concn. ( $> 6\%$ ) of oxidising agent, and then only after 20 hr. at 60° C. and pH 5. The oxychlorose formed resembles the alkaline hypochlorite type. Cotton cellulose is not attacked under ordinary industrial bleaching conditions. G. L.

**Hydrogen Peroxide Bleaching of Textile Materials.** H. G. Smolens. *Amer. Dyestuff Rep.*, 41, P 575-P 577, P 580 (15 Sept. 1952).

A review in which the main emphasis is on the historical development of the cold peroxide bleach for cotton, and the white-scarf process for wool. J. W. D.

**Application of Peroxygen Compounds in the Textile Industry.** (III p. 520).

**Microscopic Investigation of Fur Bleaching and Dyeing** (VIII p. 534).

**Application of Ultrasonics to Textile Processes** (X p. 535). Chemical Changes produced in the Cellulose Macromolecule by Oxidising Agents. VII—Oxidation of Cellulose by Sodium Hypochlorite (XI p. 537).

**VIII—DYEING**

**Significance of Hydrogen Bonding for the Dyer.** W. Happe. *Reyon, Zellwolle u. Chemiefasern*, 30, 286-295 (1952); *Chem. Abstr.*, 46, 8859 (25 Sept. 1952).

A discussion (25 references) of the role of hydrogen bonding in the use of selective dyes and textile assistants on both natural and man-made fibres. C. O. C.

**Absorption of Direct Dyes by Cellulose.** W. Harrison. *Chem. and Ind.*, 758-759 (2 Aug. 1952); F. L. Usher. *Ibid.*, 808 (16 Aug. 1952).

In a letter, Harrison criticises modern theories of dyeing as recently reviewed by Standing (*ibid.*, 527-530 (14 June 1952)) on the grounds that the assumption of preferential absorption of dye anions is unnecessary and erroneous. Experimental and theoretical evidence is quoted, and the view expressed that mathematical deductions in the absence of experimental observations can be misleading and mutually contradictory.

In reply, Usher agrees that the use of unsupported mathematical deductions is unreliable, but quotes experimental evidence for the preferential absorption of coloured dye ions. A. J.

**Silicon-containing Azo Dyes. III—Dyeing Properties, Absorption Spectra, and pH Range.** S. V. Suntharkar and H. Gilman. *Text. Research J.*, 22, 574-583 (Sept. 1952).

Dyes prepared by coupling several diazonium salts with *m*-trimethylsilyl- and *m*-triphenylsilyl-dimethylamine have fairly good substantivity for cellulose acetate, nylon, silk, and wool, but their light fastness is only moderate to good. The *R*,*Si* substituent has an unfavourable effect on both substantivity and light fastness in the 6-trimethylsilyl and 6-triphenylsilyl derivatives of 2-naphthol. The *R*,*Si* substituent has no favourable effect on the pH range (interval in which change in colour occurs) of the dyes examined. A. B.

**Sorption of Orange II by Wool.** J. Kruger, R. Donovan, and P. Larose. *Canadian J. Technol.*, 30, 215-221 (Sept. 1952).

Wool is boiled in soln. of Orange II in presence of aetic and phosphoric acids, and the amount of dye sorbed is determined by colorimetric measurements of the changes in dye concn. Sorption isotherms are represented by equations similar to those previously obtained for dyeings carried out in presence of  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ ; determinations made with low dye concn. show that the complete isotherms are S-shaped and that the first portion of the curve can be represented by a Freundlich type of equation. From consideration of the internal surface area of the micelles, it is suggested that, when large amounts of dye are adsorbed, the dye molecules lie within the fields of attraction of one another. J. W. B.

**Effect of Copper in Wool Dyeing.** L. F. Story. *J.S.D.C.*, 68, 512 (Dec. 1952).

**Controlled Vat Dyeing. I—Stabilisation of Vat Dyes at Elevated Temperatures.** C. L. Zimmerman, J. M. Mecco, F. Fordenwalt, and T. F. Cooke. **II—Instrumental and Analytical Measurement.** G. L. Royer.

**III—Leuco Potentials.** E. E. Linkeen, S. Grand, and F. Fordenwalt. *Amer. Dyestuff Rep.*, 41, 597-601, 601-608, 608-610 (29 Sept. 1952).

I—It is often expedient to raise the temp. of application of normal (non-solubilised) vat dyes to obtain levelness, particularly in continuous processing, and in package dyeing; many of these dyes are thereby damaged, suffering over-reduction, hydrolysis, or molecular rearrangement, amongst other things. Stabilisation of the leuco form by adding dextrin to the dyebath is effective but very wasteful. Amongst compounds more recently found to protect the leuco forms of vat dyes are inorganic nitrites and the hydroxyamines. Reaction between  $\text{NaNO}_2$  and  $\text{Na}_2\text{S}_2\text{O}_4$  at elevated temp., in concn. normal for jig dyeing, is negligible, but at higher concn., as used for continuous dyeing, reaction is rapid. This reaction is suppressed by addition of  $\text{Mg}$  or  $\text{Mn}$  salts, or triethanolamine, to the dyebath, and by this means it is possible to dye satisfactorily at temperatures up to  $200^\circ\text{C}$ , without over-reducing the dye. These latter additions do not in themselves materially affect the stabilisation of the leuco compound. A kinetic mechanism is proposed to explain the protective action of  $\text{NaNO}_2$ . Other stabilisers include chlorates, bromates, iodates, nitroparaffins, and aromatic nitro and nitroso compounds.

**II**—(i) The concn. of fine dispersions of oxidised vat pigments may be found spectrophotometrically; Beer's law is closely obeyed. (ii) Spectrograms of oxidised and leuco forms of a dye usually differ materially, and spectral data taken at regular intervals allow reduction rates to be calc. (iii) The stability of leuco compounds in dyebaths of commercial concn. at elevated temp. may be studied by means of the Dyecometer. (iv) Instrumental and test paper methods are described for the measurement of leuco potentials. (v) The value and methods of pH control are briefly dealt with. (vi)  $\text{Na}_2\text{S}_2\text{O}_4$  may be determined by indigo titration, iodometry, ferricyanide titration, absorption of atmospheric oxygen ("vat-ometer"), or polarography, providing other reducing agents are absent or are allowed for; end points in the first three of these may be detected potentiometrically. (vii) The estimation of  $\text{NaNO}_3$  by its reaction with sulphamic acid is discussed, and a method described in which the nitrogen liberated is measured in a simple gas burette, this being claimed to give reliable results.

**III**—The potential of a Pt electrode in a vat depends more on concn. of  $\text{NaOH}$  or of  $\text{Na}_2\text{S}_2\text{O}_4$  than on concn. of  $\text{Na}_2\text{S}_2\text{O}_4$  and does not, therefore, measure the reducing power of the bath. Leuco potential of a dye is the potential above which dye is completely reduced, or below which the leuco form begins to oxidise and ppt. A fall in pH may alter the leuco potential of a dye, but if it is ensured that the  $\text{NaOH}$  concn. is sufficient, the dyer may greatly economise in  $\text{Na}_2\text{S}_2\text{O}_4$  consumption by using small additions of  $\text{NaOH}$  and  $\text{Na}_2\text{S}_2\text{O}_4$  to maintain a potential slightly above the leuco potential value. By this method light shades have been dyed at 200 °F. with an  $\text{Na}_2\text{S}_2\text{O}_4$  consumption of ~0.1 oz./gal (U.S.A.). The experimental determination of leuco potentials is described, and values for 40 dyers at 140 °F. and 205 °F. are given. J. W. D.

**Turkey Red Dyeing in Scotland—Its Heyday and Decline.** R. A. Peel. J.S.D.C., **68**, 496-505 (Dec. 1952).

**Electrochemical Measurements in Pyridine. II—Redox Potentials of some Quinones.** A. K. Gupta. J.C.S., **3479**-3483 (Sept. 1952).

The technique developed in Part I (J.C.S., **3473** (Sept. 1952)) for potentiometric measurements in anhydrous pyridine is now used to determine the redox potentials in that solvent of twelve quinones (including higher polycyclic quinones) and of indigo. In some cases the entropies of reduction have also been determined. Sufficient agreement has been found between calculated and measured values to show that the empirical relations used to predict redox potentials are of considerable value, but appreciable discrepancies exist, and the possible reasons for these are discussed. A very large semiquinone formation constant has been found for anthraquinone, whose semiquinone is only feebly coloured (pale brown). H. H. H.

**Dyeing Acetate Rayon. V—Dyeing with Indigo.** R. Tanaka, K. Seko, and T. Murayama. *J. Soc. Textile Cellulose Ind. (Japan)*, **8**, 124-126 (1952); **VI—Dyeing with Indigo (Dispersion).** R. Tanaka and K. Seko. *Ibid.*, 192-194. **VII—Dyeing with Indigo (Mechanism).** R. Tanaka and T. Murayama. *Ibid.*, 194-196. *Chem. Abstr.*, **46**, 8858 (25 Sept. 1952).

V—Acetate rayon immersed in an indigo bath (alkali completely neutralised with acetic acid) at 39 °C. for 0.5-32.0 hr. was dyed more deeply as time went on by progressive absorption of indigo by the fibres.

VI—The dispersion of indigo in 6.2 g./150 c.c. solution by adding acetic acid (estimated by diffusion through 5% gelatin gel) was the less the more acid was added, the indigo forming larger particles. Such particles, if not too large, have less surface area and should dissolve in the acetate rayon slowly, but they dye rapidly because dyeing seems to depend mainly upon stability of the dye and not upon its solubility.

VII—Indigo solution neutralised with acetic acid was shaken with ethyl acetate at 11-14°C. for 10-160 min. The amount of indigo extracted by the solvent increased linearly with the amount of acetic acid added, and the solubility curve became flatter with longer shaking. Thus acetic acid affects the speed at which the indigo is dissolved and not its solubility. From this it would seem that the affinity of indigo for acetate rayon can be explained by the solid solution theory. C. O. C.

**Dyeing Plain and Face-finished Auto Fabrics.** M. G. Morris. *Amer. Dyestuff Rep.*, **41**, P 479-P 481 (4 Aug. 1952).

Best-quality car-lining fabrics contain up to 70% wool with a balance of nylon or rayon. A finishing process is described which includes milling, scouring, napping, pilling, drying, shearing, pressing, and decanting. Dyes fast to light and pilling must be used. The effects of lime from pulped wool are discussed, and methods given to prevent them. A. H.

**Problems presented by the New Fibres (Identification and Dyeing).** H. P. Baumann. *Amer. Dyestuff Rep.*, **41**, P 453-P 456 (21 July 1952).

A discussion of the dyeing problems presented by the new fibres. Dyeing procedures for Dacron, dynel, Acrilan, Orlon, and nylon are given, including the use of swelling agents and the cuprous ion technique. A small section devoted to fibre identification includes a staining method which employs a mixture of equal proportions of Anthraquinone Blue SWF, Chromorhodine BR, and Celliton Fast Yellow GA: 0.5 g. is dissolved in 200 ml.  $\text{H}_2\text{O}$  and boiled; 1 ml. of 50%  $\text{CH}_3\text{COOH}$  is added, and the fibre sample is boiled for 2-3 min. and rinsed at 120 °F. The resulting colours are—nylon, dark green; Acrilan, grey; dynel, red-brown; Orlon, pink; and wool, black. A. H.

**Dyeing Dynel and Related Products.** T. A. Field. *Amer. Dyestuff Rep.*, **41**, P 475-P 478 (4 Aug. 1952).

A discussion of the present state of knowledge regarding dyeing acrylic fibres. Basic, acid, and vat dyes are applicable at 250 °F. in pressure dyeing machines, but in practice dynel is dyed at 205 °F. with standard equipment using acetate rayon dyes, acid dyes, or a mixture of both. The affinity of acid dyes at 205 °F. is increased by the addition of *p*-phenylphenol, or, in the case of heavy dyes, by *p*-phenylphenol and cuprous ions developed in the dyebath from  $\text{CuSO}_4$  and a reducing agent, which may be either zinc formaldehyde-sulphonate or hydroxylammonium sulphate. Cuprous copper is specific to acrylonitrile and is believed to form a bond between nitrile group and dye molecule, producing dyes of high washing fastness. Controlled addition of Cu by a "drip" method eliminates precipitation of Cu in the dyebath. A. H.

**Dyeing Orlon.** E. Szlosberg. *Amer. Dyestuff Rep.*, **41**, P 510-P 516 (18 Aug. 1952).

Orlon can be dyed with comparative ease when reduced copper is present in the dyebath. The cuprous ion technique first used for dyed becomes less effective as the proportion of acrylonitrile to other polymer increases; thus Orlon (approx. 100% acrylonitrile) is not dyed as effectively as dynel (approx. 40% acrylonitrile). Different types of Orlon exhibit different rates of absorption, which depend on the acrylonitrile content and the degree of orientation; thus Orlon 81 (all filament fibre) does not dye as well as Orlon 41 (staple fibre). Acid dyes give the best results and have good affinity for Orlon 41; few, however, are suitable for producing heavy dyes on Orlon 81, and in this case the use of a swelling agent such as phenol or benzoic acid is recommended. The dyeing technique involves the addition to the dyebath of  $\text{CuSO}_4$  and hydroxylammonium sulphate. Problems of copper precipitation in the dyebath have been eliminated by using a "drip method", wherein the  $\text{CuSO}_4$  and the reducing agent react separately, and are added to the dyebath over a period of 30 min. Improved exhaustion and better penetration are produced by this method. Absorption of Cu is dependent on pH, which should be 2-3, temp. (212 °F.), and duration of dyeing, which must be prolonged for the best results. A. H.

**Variables connected with the Dyeing of Orlon.** G. D. Fronmuller. *Amer. Dyestuff Rep.*, **41**, P 578-P 580 (15 Sept. 1952).

Discussion relates to the cuprous ion method of applying acid dyes to Orlon. Presence of one acidic group in the dye molecule is essential, but more than one impedes penetration of the dye. The reducing agent must have a sufficiently high redox potential to reduce  $\text{Cu}^{2+}$  to  $\text{Cu}^{+}$ , but without affecting the dye. Rate of dyeing is materially affected by the accompanying anions in the dyebath— $\text{Cl}^-$  retards dyeing,  $\text{SO}_4^{2-}$  has no effect even when in excess, whilst  $\text{PO}_4^{3-}$  increases the dyeing rate by one-third. J. W. D.

**Surface Films of Bromophenol Blue.** H. C. Saraswat.  
*Kolloid Z.*, **127**, 124–129 (July 1952).

Surfaces of Bromophenol Blue show time-dependent surface tension. An initial fall takes place rapidly, but is followed by a slower, further decrease. This indicates an electric potential barrier (double layer) coming into play, slowing down the entry of fresh molecules into the surface after the first have built up the potential. This delaying effect is greater when the solution has higher pH values; the pH effect is reduced by neutral salts. At low pressures, the force-area curves show the film to be two-phased; at high pressures it is single-phased. L. P.

**Surface Films of Bromothymol Blue.** H. C. Saraswat and A. Kalyanasundaram. *Kolloid Z.*, **127**, 129–131 (July 1952).

Force-area curves of films of Bromothymol Blue show that the surface film is single phased and has the same structure at similar pressures even for different concentrations in the solution. Data for the rate of accumulation in the interface are also given. L. P.

**Solubilisation of Iron and Chromium Hydroxides.**  
E. H. Daruwalla and G. M. Nabar. *Kolloid Z.*, **127**, 33–38 (June 1952).

Iron and chromium hydroxides can be peptised to give a clear, stable solution if sufficient non-electrolyte such as sugar is present before the NaOH is added. More than the theoretical alkali must be added to prevent precipitation. With increasing NaOH concn. the minimum amount of sugar necessary decreases to a limiting value. Glycerol cannot peptise ferrous hydroxide, but chromium is solubilised by it even up to considerable concentrations. Ferrous hydroxide is, however, strongly peptised when chromium also is present. The minimum amount of NaOH and sugar necessary to peptise a given quantity of ferro and chromium hydroxides when together is greater than the sum of the individual amounts. The hydroxides were more stable to dialysis when formed together than when separate, which indicates complex formation. L. P.

**Sensitivity to Lime of Anionic Leather Dyes.** G. Otto.  
*Leder.*, **2**, 210–213 (1951); *Chem. Abstr.*, **46**, 5876 (25 June 1952).

The effect of lime in hard water used for dyeing was tested by passing CO<sub>2</sub> into a dispersion of lime and homogenising to yield a dispersion of CaCO<sub>3</sub> containing 74 g. CaO per litre. One c.c. of the dispersion and 10 c.c. of dye liquor (containing 0.6–10<sup>-3</sup> M pure dye) were mixed and left overnight, after which the dye concn. was measured colorimetrically. Acid dyes were affected only slightly or not at all, but many direct cotton dyes were completely precipitated. In general a dye reacted with CaCO<sub>3</sub> if it contained (1) a nucleus with two attached NH<sub>3</sub><sup>+</sup> groups but no SO<sub>3</sub><sup>2-</sup> group, (2) an aromatic nucleus having two OH groups, or (3) a naphthol nucleus with an amino group. A carboxyl group was very effective, especially if *ortho* to an OH group. C. O. C.

**Sensitivity to Lime of Anionic Dyes.** K. Eitel. *Leder.*, **3**, 56–58 (1952); *Chem. Abstr.*, **46**, 5876 (25 June 1952).

Otto's method (above) measures only stability to temporary hardness, whereas stability to permanent hardness is much more important. This can be measured by diluting 10-c.c. samples of dye liquor (0.02 g./litre) with water and with solutions of permanent hardness 10–40° (Tierman hardness). The sample is examined in bright light for precipitate after 1 or 24 hr. Change in strength of hue of the sample is important. The structure of a dye is no reliable guide to its stability to hard water. C. O. C.

**Microscopic Investigation of Fur Bleaching and Dyeing.** F. Stather. *Gas, Abhandl. deut. Lederinst.* *Freiberg Sa.*, (8), 3–11 (1952); *Chem. Abstr.*, **46**, 7805 (25 Aug. 1952).

Killing with 2% NaOH destroys the medulla of white rabbit hairs, but the cortex is not visibly affected. With black hairs destruction of pigment granules is incomplete. Successive treatment with Na<sub>2</sub>CO<sub>3</sub>, FeSO<sub>4</sub>, and alkaline H<sub>2</sub>O<sub>2</sub> destroys the muscular pigment completely in 12 hr., while pigment granules in the cortex change from blue to yellow. In rabbit hairs treated successively with Na<sub>2</sub>CO<sub>3</sub> and Ursol D + H<sub>2</sub>O<sub>2</sub>, the cortex is faintly tinted, but if the hairs are first treated with NaOH and Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, the entire cortex is intensely coloured. Further tests with lime,

white wool hairs, without medulla, show that dye penetration and intensity of colour can be varied by mordanting. Hairs mordanted with 0.25% Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> show complete pale dye penetration, while 2.5% Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> resulted in intense coloration of the outer third of the cross section. Hairs mordanted with 0.25 or 2.5% FeSO<sub>4</sub> showed complete dye penetration and low colour intensity. 0.25% CuSO<sub>4</sub> mordant gave intense surface dyeing, while 2.5% gave complete penetration. Pickling and tanning had no visible effect on microstructure, but Cr and Al tans resulted in slightly deeper dyings. C. O. C.

**Adsorption of Dyes on Hydrated Aluminium Hydroxide.** S. N. Tewari. *Kolloid Z.*, **128**, 19–22 (Aug. 1952).

The adsorption of Methylene Blue or Orange II by aluminium hydroxide of different degrees of basicity is studied. Previously (*ibid.*, **124**, 31 (1951)) colloidal solutions were used; here solid precipitates are examined. The results are similar in that more acid dye is taken up by the more basic hydroxide, and more basic dye by the less basic. Ageing and increase of temperature decrease the amount adsorbed, as before. L. P.

PATENTS

**Metachrome Dyeing.** Victor Chemical Works.

*USP* 2,593,850

Use of a mixture of diammmonium phosphate and ammonium sulphate enables the bath to be alkaline at the start and to attain pH 5.2–5.8 on boiling. C. O. C.

**Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Polymers or Copolymers of Conjugated Dienes.**  
N.V. De Bataafsche Petroleum MS. *BP* 680,491

Dyes, etc., are incorporated in e.g. filaments of natural or synthetic rubber which has been treated with SO<sub>2</sub>, by immersing them in presence of swelling agents in aqueous solutions or dispersions of the dye, etc. C. O. C.

**Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Macromolecular, Poly-unsaturated Compounds.**  
N.V. De Bataafsche Petroleum MS. *BP* 680,492

Amendment of *BP* 680,491 (above). The process is made continuous by extruding a solution of a macromolecular unsaturated compound into a solution of sulphur dioxide and then passing the resultant filaments into an aqueous solution or dispersion of the dye or other substance to be applied. C. O. C.

**Coloured Glass Fibre Cloth.** Owens-Corning Fiberglas Corp. *USP* 2,593,817

Glass fibre fabric is treated with an organic compound, preferably a salt, of a metal (having a colouring oxide) mixed with an organosilicon liquid in which the organic compound is soluble or with which it is compatible in organic-solvent solution, and heated for 1–2 sec. at 1200–1250° F. to 1–5 min. at 600° F. This imparts permanent coloration and relaxes the glass fibres, so that the treated fabric may vary in flexibility from starchy stiffness to silky softness while being supple and crease-resistant.

*USP* 2,593,818

The material is coated with a finely divided colouring metal oxide and heated for from 2 sec. at slightly below m.p. to 5 min. at 600° F. This gives permanent coloration with improvement in handle. C. O. C.

Minimising "Mottling" with Direct Cotton Dyes in Paper Coloration (*Manufacturers' Publications* p. 517).

Application of Peroxygen Compounds in the Textile Industry (III p. 520).

Recent Researches on Wool (VI p. 529).

Application of Ultronics to Textile Processes (X p. 535). For Improvement, also Aniline Dyes and Synthetic Fats for the Leather Industry (XII p. 528).

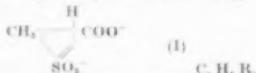
IX—PRINTING

**Photodecomposition of Aqueous Solutions of Diazonium Salts in the presence of Mercurous Ions.** J. G. Bos, R. J. H. Alink, and C. J. Dippe. *Rec. Trav. chim.*, **71**, 945–953 (Aug. 1952).

Irradiation of dilute solutions of *p*-aminobenzenediazonium salts and mercurous nitrate gives almost colourless solutions, which slowly turn brown and deposit metallic mercury without the formation of mercuric ions, the reaction being retarded by nitric acid. Irradiation of

a dilute solution of 3-diazo-2-hydroxy-1-methylbenzene-5-sulphonic acid and mercuric nitrate made slightly acid by nitric acid rapidly gives a quantitative transformation into metallic mercury and the mercuric compound of the photodecomposition product in equimolecular proportions, the reaction not being inhibited by the presence of nitric acid.

The former reaction appears to be a reduction of the mercurous nitrate by the photodecomposition product of *p*-aminobenzenediazonium salts, viz. *p*-aminophenol, while the latter reaction can be explained by disproportionation  $Hg^{2+} \rightarrow Hg + Hg^{+}$ , removal of the  $Hg^{+}$  by the decomposition product (I) of the *o*-hydroxy-diazonium salt displacing the equilibrium to the right.



**Quantum Yield of the Photodecomposition of some Aromatic Diazonium Salts.** J. de Jonge, R. Dijkstra, and G. L. Wiggerink. *Rec. Trav. chim.*, **71**, 846-852 (Aug. 1952).

The quantum yield of the photodecomposition of diazo compounds of *p*-aminophenylamine, some aminophenols and aminonaphthols, and 2-methoxyaniline-5-sulphonic acid has been determined by irradiating a solution of the diazonium compound with monochromatic light of 3650 Å, and measuring the volume of nitrogen evolved. The quantum yield of photoisomerisation of the stable (*trans*) form *p*-methoxybenzenediazonocyanide in the presence of silver nitrate has also been determined. C. H. R.

**Effect of Steaming on Textile Fibres.** R. Haller. *Textil-Rund.*, **7**, 359-365 (Aug. 1952).

The effects of steaming by vapours of varying composition were studied. Vapours examined comprised steam to which had been added acetic acid, formic acid,  $NH_3$ , formaldehyde,  $SO_2$ ,  $SO_3$  and formaldehyde,  $Cl_2$ ,  $Br_2$ ,  $ClO_2$ ,  $NH_4Cl$ , and  $O_2$ . The results are critically discussed and their significance in printing processes is indicated. G. L.

PATENTS

**Adhesives for Fixing Fabrics to Printing Tables.** Lankro Chemicals. *BP* 680,272

A polymer of an alkyl methacrylate (Alk of 6-12 C) or a copolymer of such a methacrylate with a lower alkyl acrylate or methacrylate (Alk of < 6 C) provides a permanently tacky surface, which enables many pieces to be printed without renewing the adhesive film. As it is water-repellent, little printing colour sticks to the table, the washing of which is greatly facilitated. C. O. C.

**Coloured Filter Layers for Photography.** ICI. *BP* 680,631

Suitable selection of a non-diffusing yellow azo dye and, if desired, of the subst. *p*-phenylenediamine, so that a soluble azomethine or indophenol dye is formed during development, allows complete removal of the yellow dye from a filter layer. C. O. C.

**2:4-Diamino Developer in Colour Photography.** General Aniline. *USP* 2,596,926

Addition of a small quantity of a pyridine derivative to a 2:4-diamino developer for silver halide emulsions in presence of a colour former results in (1) more energetic development of positive silver halide, (2) increased dye formation efficiency, and (3) more brilliant dye images. C. O. C.

Coloured Glass Fibre Cloth (VIII p. 534).  
Dye Contamination Detector (XIV p. 543).

**X—SIZING AND FINISHING**

**Sizing of Rayon Warps with Gelatin.** M. Martin. *Bull. Inst. textile France*, (33), 33-38 (June 1952).

A discussion of practical details with special emphasis on the subsequent desizing process. Two recipes are included. G. L.

**Sizes for Glass Textiles for Reinforcing Polyester Plastics.** L. P. Bielefeld and T. E. Philipp. *Amer. Dyestuff Rep.*, **41**, 501-506 (18 Aug. 1952).

An outline of the operations for converting molten glass into fibrous forms used for reinforcing plastics. The requirements of a yarn size for this purpose, procedures

for evaluating sizes, and comparisons of the handle and performance characteristics of glass fibres treated with various sizes are given. A. H.

**Some Aspects of the Drying and Heating of Textiles.**

**VII. The Stability of Thermal Modifications of Fibres in Subsequent Treatments.** J. M. Preston and S. P. Gundavada. *J. Soc. Dyers Colourists*, **68**, 511 (Dec. 1952).

**Improvement of Fibre Qualities by Aftertreatment.**

**I—Crease Resistance of Rayon.** H. Sobe, K. Matsukage, and K. Murakami. *J. Soc. Textile Cellulose Ind. (Japan)*, **8**, 241-244 (1952); *Chem. Abstr.*, **46**, 8860 (25 Sept. 1952).

Creasing of rayon seems to be due not only to the arrangement of microfibrils but also to the elastic fatigue (expressed by the time of crease retention under a load) and particularly to the moisture content. Aftertreatment, e.g. with resins, decreased the moisture content and hence increased crease resistance. C. O. C.

**Synthetic-resin Treatment of Rayon. III—Relation between Synthetic Resin Content and the Physical Properties of the Filaments.** K. Migita. *J. Soc. Textile Cellulose Ind. (Japan)*, **8**, 70-72 (1952); *Chem. Abstr.*, **46**, 8860 (25 Sept. 1952).

Impregnation with even a small amount of bis(hydroxymethyl)resin results in less crimping, marked reduction in transverse swelling in water or dil. aq. NaOH, and lower regain. C. O. C.

**Urea Formaldehyde Fixation on Viscose Rayon.**

R. F. Nickerson. *Amer. Dyestuff Rep.*, **41**, P 482-P 486 (4 Aug. 1952).

A study of the wash fastness of urea-formaldehyde resin on viscose rayon is reported. Resin retention, determined by weight loss after acid stripping, can vary with the amount of resin originally applied, and does not give a precise measurement. There is no close correlation between retained resin and fabric shrinkage, and since the purpose of applying the resin is to produce desirable fabric stabilisation, a direct measure of fabric properties is recommended to evaluate these finishing processes. The author therefore agrees with Foulds, Marsh, and Wood, who originally suggested that "the success of the treatment is to be judged by physical rather than chemical tests". A. H.

**Stabilisation and Shrinkage Control.** W. A. Wardell. *Amer. Dyestuff Rep.*, **41**, P 546-P 549 (1 Sept. 1952).

The effect and efficiency of processes for producing stabilisation and shrinkage control of textile fabrics are discussed. All the known methods are tabulated with the degree of efficiency achieved for each property desired. It may be necessary to obtain characteristics in a finished fabric which are not all produced by one process, in which case modification of the process is made or a combination of two finishing processes used. A. H.

**Treatment of Wool with Sodium Chlorite.** C. Earland and K. G. Johnson. *Text. Research J.*, **22**, 591-598 (Sept. 1952).

It is shown that the simplest agents for the activation of neutral chlorite solutions so that they may be used to shrink-resist wool are formaldehyde and certain sulphur-containing reducing substances. The advantages of this treatment over the acid chlorite process are that processing may be carried out under neutral conditions at room temperature and that the wool suffers very little damage. The mechanism of the reaction, using formaldehyde as activator, is investigated. A. B.

**Application of Ultrasonics to Textile Processes.** A. A. de Araujo. *Amer. Dyestuff Rep.*, **41**, P 615-P 619 (29 Sept. 1952).

The physical and chemical effects of ultrasonic vibrations upon materials in general, and cellulose fibres in particular, are discussed. Applications (both actual and potential) in the textile finishing field include—(i) to increase dyeing rate, improve penetration, and allow the use of lower dyeing temp.; (ii) in artificial fibre production, to control uniformity and re-orientation of crystallites in regenerated cellulose, to accelerate coagulation of viscose filaments leaving the spinneret, in the continuous clarifying of the viscose setting bath, and in the degassing of viscose soln.; (iii) in laundering and cleaning, removing graphite from nylon, and cleaning raw cotton and wool; (iv) to prepare highly dispersed pigment pastes and finishes; (v) in testing the dynamic properties of filaments,

yarns and cords, and (v) in reducing the tendency of wool to felt and shrink. Lack of suitable ultrasonic apparatus does not yet permit a transition from laboratory to plant scale. There are 44 references to the literature, and an additional bibliography of 26 items.

J. W. D.

**Non-felting Wool and Wool Mixtures.** F. C. Wood  
J. S. D. C. 68, 485-495 (Dec. 1952).

**Shrinkproofing Wool Goods. III. Treatment with Thiourea Formaldehyde Resin and Change of Elasticity of Wool.** N. Azuma, F. Bekku, and T. Ito.  
J. Soc. Textile Cellulose Ind. (Japan), 8, 127-130 (1952); Chem. Abs., 8861 (25 Sept. 1952).

The elasticity of wool in water changed only slightly when thiourea-formaldehyde resin was deposited on the fibre surface, but decreased markedly when the resin penetrated into the fibre. In the former case the effect on felting power was slight, but in the latter case it was dominant.

C. O. C.

**Crimping Crimpless Keratinous Fibres. I. Crimping Angora Rabbit Hair by means of Fuming Nitric Acid Vapour.** M. Oku and I. Sakaguchi.  
J. Soc. Textile Cellulose Ind. (Japan), 8, 73-74 (1952);  
II. Crimping Angora Rabbit Hair by means of Neutral Salt Solutions. Ibid., 224-226; Chem. Abs., 46, 8859 (25 Sept. 1952).

I. Angora rabbit hair kept at room temperature in a desiccator containing fuming nitric acid vapour became yellowish and crimped in 25 hr. The crimp increased on longer exposure, but strength and elongation, which had increased up to 25 hr., decreased thereafter.

II. Crimping was produced without loss of strength by immersing for 15 min. in a bath of 18%  $\text{Ca}(\text{NO}_3)_2$  diluted 40 times at 102°C. or a similar bath of  $\text{CaCl}_2$  at 103.5°C. for 50 min.

C. O. C.

**Crimping of Amilan.** I. M. Watanabe. J. Soc. Textile Cellulose Ind. (Japan), 8, 218-220 (1952); II. Ibid., 220-224; Chem. Abs., 46, 8859 (25 Sept. 1952).

I. Amilan (polyacrylamide) filaments were effectively crimped by first stretching them three times at low temperature, then treating at 90°C. for 70 min. in 37% formalin containing 1 g.  $\text{NH}_4\text{Cl}$  and finally, after cooling, immersing for 5 min. in 59.61%  $\text{H}_2\text{COOH}$ , 27.29%  $\text{H}_2\text{SO}_4$ , 17.18%  $\text{HNO}_3$ , or 9.13%  $\text{HCl}$ . The crimp ratio was 1.7-2.2, crimp retention practically 100%, and crimp recovery from stretching 94-100%.

II. Amilan can be crimped also by treating with formalin (as above), immersing in a mild swelling agent (water, methanol, or acetone) at 130°C. for 80 min., then either suddenly cooling with water or immersing for 0.25-0.5 sec. in paraffin wax at 145-160°C., and finally cooling rapidly with water. The finer the filament the better the crimping. The crimps can be fixed by boiling in water for 90 min.

C. O. C.

#### PATENTS

**Permanent Embossed Effects on Regenerated Cellulose Fabric.** Betex Sales Corp. U.S.P. 2,593,207

Embossed effects fast to washing and improved tensile strength, body, and handle are obtained by (1) impregnating with a 1-3% solution in an aliphatic alcohol of an *N*-alkoxymethylpoly(allyl amide) resin and an acid catalyst, so that the fabric takes up 60-100% of its weight of the liquor, (2) drying at < 250°F. to leave 10-20% moisture content, (3) embossing at 250-375°F., and (4) baking at 250-350°F. for 15-4 min.

C. O. C.

**Reducing the Felting Properties of Wool.** American Cyanamid Co. B.P. 679,386

The material is impregnated with an acidic colloidal aqueous solution of a polymerised, positively charged, aldehyde condensate of an aminotriazine having > 1 amino group and an  $\text{H}$  attached to each amino  $\text{N}$ . The solution contains 0.5-7.0 mol. of a water-soluble, sat. aliphatic monocarboxylic or hydroxymonocarboxylic acid (of dissociation constant  $1.4 \times 10^{-3}$  to  $2.5 \times 10^{-4}$ ) for each mol. of monomeric aminotriazine aldehyde condensate from which the polymerised product has been formed. The material should take up 5-15% on its dry weight of the polymer. There is no impairment of handle, tensile strength, or wearing qualities.

C. O. C.

**Shrinking Fabrics.** H. Haas. B.P. 679,442

The fabric, if not already wet from previous processing, is treated with saturated but dry steam so as to swell the

fibres, and is then passed into cold or warm damp air while relaxed. Suitable machinery is described.

C. O. C.

**Coated Fabrics.** American Viscose Corp.

U.S.P. 2,593,553

An open mesh fabric is coated without impregnating its component yarns by applying to each side of the fabric a thin continuous non-self-supporting impervious film of an organic thermoplastic material supported on a temporary backing. The assembly is then hot pressed to cause the films to adhere to the fabric and to fuse to each other in the spaces between the yarns of the fabric. The backings are then removed from the films.

C. O. C.

**Joining Calender Guide Tapes.** HrC. B.P. 680,764

Ribon for use as a laundry calender tape is made either by ribbon weaving or by slitting a 36 in. wide fabric, the yarns being of tenacity > 6 g. denier. The ribbon, or fabric prior to slitting, is passed through a 6% (by wt.) soln. of cellulose acetate in acetone, and wiped so as to leave 8-10% by wt. of cellulose acetate adhering to the material; after removing the acetone the tapes are passed through the calender, and their ends joined in a similar manner with 6% cellulose acetate soln. A smooth joint is produced with no troublesome lumps or ridges.

J. W. B.

Action of Bromine Water on Wool and other Animal Hairs (VI p. 529).

Significance of Hydrogen Bonding for the Dyer (VIII p. 532).

Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Polymers or Copolymers of Conjugated Dienes (VIII p. 534).

Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Macromolecular, Poly-unsaturated Compounds (VIII p. 534).

Effect of Steaming on Textile Fibres (IX p. 535).

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

**Heats of Wetting of Polysaccharides. II—Relation between Adsorption Isotherms and Heats of Wetting.** T. Nakagawa. Bull. Chem. Soc. Japan, 25, 171-174 (June 1952).

Net heats of adsorption, defined as (heat of adsorption) minus (heat of condensation), have been measured for many polysaccharides at various R.H., and it is suggested that, when  $\text{R.H.} < 80\%$ , the net heat of adsorption is proportional to the amount of water adsorbed at the same R.H.

A. J.

**Colloidal Properties of Cellulose Micelles.** B. G. Rånby. Discussions Faraday Soc., (11), 158-164 (1951).

When cellulose is hydrolytically depolymerised by boiling with 2.5%  $\text{H}_2\text{SO}_4$ , subsequent washing with dist. water causes peptisation, which begins when the pH reaches 3.3-5, or 10-10.5 if the wet, washed hydrocellulose is first immersed in 0.1% NaOH. By this method, using a preparative centrifuge (field  $\sim 1000 \text{ g}$ ), sols containing up to 0.5 g. cellulose per 100 ml., and with the particles negatively charged, have been prepared from native and mercerised wood cellulose, and from viscose rayon; they are stable within the pH range 3.5-10 for several days. The sols contain both free and aggregated micelles, resolvable on electron micrographs, which are bundles of 100-150 cellulose molecules. Their width is  $\sim 70 \text{ \AA}$ , and their length corresponds to the chain length calc'd. from the degree of polymerisation; these dimensions agree with those previously calc'd. by Hengstenberg and Mark from the X-ray diagrams. The sols are coagulated by small amounts of electrolyte ( $0.2-0.3 \times 10^{-3} \text{ M}$  NaCl or  $0.015-0.020 \times 10^{-2} \text{ M}$   $\text{La}(\text{NO}_3)_3$ ), and are thus hydrophobic. Of the sols prepared, those from rayon were the least electrolyte-sensitive at high pH; all were less sensitive to multivalent ions than hydrophobic sols such as those of  $\text{Au}$ ,  $\text{Ag}_2\text{S}$ , and  $\text{MnO}_2$ . The sols may be protected against coagulation with NaOH by adding  $\gamma$ -cellulose, this expedient being least effective with the sols prepared from viscose rayon. Ultrasonic irradiation causes the breakdown of secondary aggregation, but so far it has not proved possible to bring about complete disintegration into free micelles.

J. W. D.

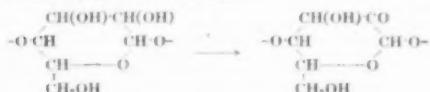
**Acid Strength of Glucuronic Acid in Comparison with that of Oxycelluloses.** P. Hirsch. *Rec. Trav. chim.*, **71**, 999-1006 (Sept.-Oct. 1952).

Interconvertibility of glucurone and glucuronic acid in dil. aq. soln. is investigated, and from the neutralisation curves and constants of free glucuronic acid compared with those of COOH groups in oxycelluloses, it is found that between pH 6 and 9 the conversion proceeds with moderate velocity, but is more rapid at pH > 9, and slow to very slow at pH < 6, when the free acid is so stable that partial conversion only occurs in strong mineral acid on heating. Oxycelluloses show a behaviour in titration analogous to that of glucurone, and the presence of lactone or acid anhydride linkages in them is therefore assumed. It is remarkable that the linking of glucuronic acid groups to chain molecules causes only a slight change in the dissociation constant.

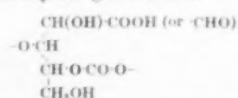
H. H. H.

**Chemical Changes produced in the Cellulose Macromolecule by Oxidising Agents. VII - Oxidation of Cellulose by Sodium Hypochlorite.** E. D. Kaverzneva, V. I. Ivanov, and A. S. Salova. *Izvestiya Akad. Nauk S.S.R. Otdel. khim. nauk.*, **751**, 762 (July-Aug. 1952).

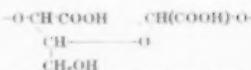
Cotton, purified under mild conditions, is oxidised with cold 0.05 N NaOCl at pH 4.6, 6.8, or 11, until a predetermined amount of oxygen, within the range 5-15 O per 100 glucose residues, has been consumed. The degree of polymerisation of the product is determined viscometrically, and its chemical nature is explored by the aid of various specific quantitative reactions. The rate of reaction is greatest in neutral soln. In general, the carboxyl content increases steadily as oxidation proceeds, and the content of ketone groups rises, particularly in the earlier stages. Under acid or neutral conditions, there is a progressive increase in the aldehyde content, and the "uronic" carboxyl content is greater than the "total" (owing to formation of lactone and carboxylic ester linkages (see *J. S. D. C.*, **68**, 329 (Aug. 1952)); but under alkaline conditions no CHO groups are formed, the uronic carboxyl content is less than the total, and the extent of carboxyl formation is much greater than in an acid or neutral treatment of equivalent oxygen consumption. Alkaline oxidation, in fact, yields some non-uronic carboxylic acids - more than can be accounted for by oxidation at the ends of chains and disruptive oxidation of the pyran ring must evidently occur. It is considered that carbonic ester linkages, a small number of which are present during acid or neutral oxidation, are continually breaking down at the rate at which they are formed, with rupture of the molecule. An attempt is made to locate the CHO groups, using a method based on their selective oxidation by hypoiodite, but difficulties arise owing to partial dissolution of the preparation by the reagent; it is established, however, that some of the CHO groups are formed by oxidation of CH<sub>2</sub>OH side-chains. It is considered that, under all conditions of hypochlorite oxidation, there are two primary reactions - conversion of a CH<sub>2</sub>OH side-chain to COOH with intermediate formation of CHO, which is a very transient stage under alkaline conditions, and oxidation of a secondary alcohol group to a ketone group.



In acid or neutral medium, further oxidation of this ketone leads to rupture of the pyran ring with formation of an aldehyde or carboxyl group and of a carbonic ester linkage leading to the adjacent glucose residue -



The chain is then readily ruptured by hydrolysis at the ester link. In alkaline medium, the *o*-hydroxylketone grouping is oxidised to a diketone and further to a dicarboxylic acid -



The mechanism whereby main chain breakdown occurs during alkaline oxidation is discussed.

A. E. S.

**Reaction of Cellulose with Formaldehyde.** R. F. Nickerson. *Text. Research J.*, **22**, 554-555 (Aug. 1952).

The results of Wagner and Paesu (*ibid.*, **22**, 12 (Jan. 1952)) are discussed, and it is concluded from experiments with boric acid that both formaldehyde and cellulose exert an activating effect on boric acid, and increase its acidity.

A. B.

**Distribution of the Substituents in partly Substituted Carboxymethyl Celluloses.** T. C. Timell and H. M. Spurin. *Scandin. Papperstidning*, **55**, 700-708 (30 Sept. 1952).

In an investigation of the distribution of the substituents in a number of carboxymethyl celluloses, only one of the two adjacent secondary hydroxyl group was found to be substituted. This was ascribed to the repellent effect exerted on the negative chloroacetate ion by the negatively charged carboxymethyl group when situated in either position 2 or 3, and probably accounts for the results previously noted on periodate oxidation of various cellulose ethers containing ionised substituents. A statistical calculation of the distribution of the substituents, assuming only one of the two secondary hydroxyl groups to be available for etherification, showed good agreement with experimental results. The composition of two samples with a rather higher degree of substitution indicated some interaction between hydroxyl groups in neighbouring anhydroglucosidic units. The position of the substituents, however, will not always account for differences in the properties noticed between apparently similar carboxymethyl celluloses, and other methods need to be applied.

S. V. S.

**PATENTS**

**Wet-strength Paper.** American Cyanamid Co.

U.S.P. 2,595,935

Addition to pulp or paper of 0.1-5.0% (on dry weight) of an uncured thermosetting resin, obtained by condensing an alkylene polyamine with a halogenohydrin, imparts increased wet and dry strength.

U.S.P. 2,596,014

Use of a urea-formaldehyde resin intercondensed with a water-soluble product obtained by treating dicyandiamide with the product obtained by condensing formaldehyde in presence of acid with a primary or secondary monoamine having at least one univalent aromatic radical attached to the amino N.

C. O. C.

Minimising "Mottling" with Direct Cotton Dyes in Paper Coloration (*Manufacturers' Publications* p. 517).

Estimation of the Distribution of the Substituents in partly Substituted Carboxymethyl Celluloses (XIV p. 544).

**XII - LEATHER; FURS; OTHER PROTEIN MATERIALS****Molecular State of Water bound to the Peptide Link.**

M. Tsuboi. *Bull. Chem. Soc. Japan*, **25**, 160-164 (June 1952).

Infrared absorption measurements on a film of amilan (*poly-D-*l*-caproamide*) in the dry state and after exposure to air at 76° R.H. show an absorption band at 2.86  $\mu$ , due to water bound to the peptide groups in amilan. Absorption bands at 3-03  $\mu$ , found with proteins are considered to be due to water bound to side-chain groups.

A. J.

**Mechanism of Vegetable Tannage.** S. G. Shuttleworth. *J. Amer. Leather Chem. Assocn.*, **47**, 603-622 (Sept. 1952).

Electrovalent forces, hydrogen bonding, and adsorption aspects of tanning, titration curves of various vegetable tannins, and stereochemical considerations are discussed in a study of the mechanism of vegetable tannage.

J. W. B.

**Pigment Finisher for Leather.** F. Welsh. *Shoe and Leather Reporter*, **624**, (12) (1951); *J. Amer. Leather Chem. Assocn.*, **47**, 494 (July 1952).

The first pigment finish, made in 1890, consisted of soap, glue, sinew, umber, and ochre for application on

dark tanned wax splits. This was followed by the incorporation of various waxes and adhesives, without, however, greatly improving the elasticity of the finish. Lakes, toners, and reduced colours replaced the earth colours, new vehicles for carrying the colouring materials were developed, and various polymers and copolymers were introduced to impart elasticity to the films. The holding power of a pigment does not necessarily depend on its concentration, since other factors, such as method of manufacture of the finish, are of great importance. Toners are pure colouring agents containing no inert matter, such as paraffins, toluoidines, and lithols. Generally paraffins bleed the most, and lithols the least, while toluoidines are fastest to light. Lakes are made by precipitating the colouring agent on inert material, such as barium sulphate or aluminium hydroxide. Reduced colours are prepared by dry blending of a toner and an inert material. Dyes and pigments differ in that the former are soluble and are applied to leather in the dissolved state, while the latter are insoluble and are applied to leather suspended in a suitable liquid vehicle. These are usually vegetable oils and glycols combined with mixtures of protein syrups, cellulose gums, silica solutions, and soya proteins. In all, there are close to 100 plasticisers in use. A good pigment finish should have the following qualities: (1) Pigments should be ground to a colloidal condition, at least three times in a wetting out medium. That will minimise sedimentation. (2) Pigments must be reasonably fast to light. (3) They shall be unaffected by alkaline solutions for a long period. (4) They shall be unaffected by heat in plating. (5) Pigments should have good covering power in comparatively weak mixture without a painted appearance. (6) The pigments should be colloidal and not granular, in order to impart a smooth, silky feel. (7) They should be unaffected by atmospheric influence. The most desirable properties of a pigment finish are uniformity, applicability, adhesiveness, flexibility, elasticity. It should have a natural feel, and should be able to withstand the various shearing operations.

C. J. W. H.

#### Reactive Groups of Collagen. I—Reactions of the $\alpha$ -Amino Group of Lysine. R. L. Sykes. *J. Soc. Leather Trades Chem.*, **36**, 267–273 (Sept. 1952).

Standard hide powder is treated with nitrous acid, dinitrofluorobenzene (DNFB), sodium glyoxalate, and sodium bromacetate, the last named providing a method of carboxymethylating collagen. It is shown that not all the lysine in collagen is available to the large DNFB molecule, and that heat shrinkage decreases the availability. Treatment with Na glyoxalate blocks some of the lysine amino groups as determined by the DNFB method; it appears that the collagen aldehyde compound is to some extent decomposed by DNFB treatment. Sodium bromacetate treatment produces carboxymethylated lysine residues equivalent to  $0.22 \times 10^{-3}$  mole lysine/g. collagen. Titration curves of the modified collagens are presented, and their significance is discussed.

J. W. H.

#### Chemical and Physiological Properties of Sodium Silicofluoride as used in the Treatment of Hides and Skins. C. C. Kritzinger. *J. Soc. Leather Trades Chem.*, **36**, 217–225 (July 1952).

Sodium silicofluoride is a bacteriostatic agent which gives complete protection to hides and skins against red-heat and other types of bacterial decomposition. It is toxic or repellent to Dermestid beetles, Timid moths, and rodents, but is not poisonous to human beings. In the soak pit it helps in opening up the stock and causes hair-removal by removing interfibillar proteins. In limiting its presence during curing leads to higher tanned yields. There are sixty six references.

A. S. F.

#### Depilatory Activity of Sodium Sulphide and Related Compounds. J. M. Gillespie. *Australian J. Sci. Research B*, **4**, 187 (1951); *J. Amer. Leather Chem. Assoc.*, **47**, 493 (July 1952).

The depilatory action of sodium sulphide on sheepskin has been investigated by measuring the pull required to remove wool from skin, the swelling of wool root shafts, and the mechanical strength of wool. The rate of action is augmented by increase in temperature, pH, and concn. of sulphide in the solution employed up to 1.0 M., but is reduced by increase to higher concentrations. The reciprocal action of the hydroxyl and hydrosulphide ions in digesting keratin is explained in terms of the ionisation

of thiol groups. Solutions of 1.0 M. sodium sulphide, partly oxidised with peroxide and adjusted to pH 12.8, completely loosen wool in 18 hr. at 20°C. only if the redox potential is below 400 m.v. Salts of various oxy-acids of sulphur are less effective depilatories than sodium sulphide, the activity diminishing with increase in the number of oxygen atoms in the anion and approximately with the heat of formation. Sodium selenide and sodium telluride possess depilatory activity equal to that of sodium sulphide.

C. J. W. H.

#### Unhairing. VII—Influence of Salts added to Lime Liquors on the Swelling of Animal Skin. R. L. Stublings and E. R. Theis. *J. Amer. Leather Chem. Assoc.*, **47**, 530–547 (Aug. 1952).

The swelling of animal skin in saturated lime liquors containing various concn. of sodium chloride, cyanide, hydrosulphide, thiosulphate, sulphate, sulphite, hydroxide, and sulphide and calcium chloride has been measured. Over a limited  $[\text{OH}^-]$  range (0.045–0.055 N.) this swelling is almost a linear function of  $[\text{OH}^-]$ . Addition of any sodium salt in low concn. produces an increase in swelling, and the calcium chloride causes a decrease. An explanation of the effect of these salts is made on the basis of the Donnan membrane equilibrium, change in fibre cohesion, and dehydration. The results obtained suggest several practical applications in tannery bathhouse operations.

A. S. F.

#### Fat Improvement, also Aniline Dyes and Synthetic Fats for the Leather Industry. D. Pense. *Oester. Leder-Zeitung*, **6**, 307 (1951); *J. Amer. Leather Chem. Assoc.*, **47**, 496 (July 1952).

A lecture discussing methods for tanning and finishing furs, leather dyes, and synthetic fats. The syntan Coronat F has a bleaching action on chrome leather, with the result that dyeing is more even if it is used. Corglon GA has less bleaching action but promotes even dyeing and a "full" leather; 0.5–2% may be used before, during, or after dyeing. Dermol oil is stable and imparts mould resistance to leather.

C. J. W. H.

#### Chemistry of the Carrotting of Rabbit Fur—II. P. Huelck and H. G. Frohlich. *Textil-Praxis*, **7**, 808–812 (Oct. 1952).

Experimental work is continued (cf. *J. S. D. C.*, **68**, 331 (Aug. 1952)) on the carrottting of rabbit fur with peracetic acid, hydrosulphite, HCl, and  $\text{HNO}_3$ , the swelling properties, cysteine content, and alkali solubility being studied. The action of these substances and also other reducing and oxidising agents, alkalis, and acids is discussed theoretically. It is shown that rabbit fur will felt only when at least 30–40% of its cysteine content is disrupted. If over 75% is affected, the felting properties of the fur are decreased. No difference is shown in subsequent felting properties whether rupture of the cysteine groups is brought about by the use of reducing or oxidising agents. Although rupture of the cysteine groups is essential for felting to take place, this is facilitated also by rupture of the hydrogen bonds and peptide linkages. Regenerated protein fibres such as Vicara, Ardin, and casem fibre do not felt and do not exhibit the property of spontaneous felting. These fibres do not contain cysteine groups.

B. K.

#### PATENTS

#### Preserving Fresh Hides and Skins. V. M. Klemola. *BP* 679,387

A mixture of not  $> 97\%$  of an aliphatic dihydric alcohol of 2–5°C. and/or glycerol and/or a polymer of such glycol or glycerol and 3–15% of an aliphatic epoxide is used. The glycerol or glycerol prevents formation of a non-swelling, insoluble sheath around the collagen fibres, and the epoxide assists in rapid penetration into the deepest layers of the skin.

C. O. C.

#### Dehairing Skins. H. Zaleman. *BP* 680,784

Dehairing of skins is effected by a composition comprising calcium hydrosulphide (2–30% by wt.), lime (33.1), inert filler, e.g. kaolin, which is adjusted according to the required consistency of the "paint", and water. In one example a sheepskin with a wool count of 58 is washed, soaked, and deburred, and painted with a composition of calcium hydrosulphide (7), lime (14), kaolin (6), and water (73). It is folded in the middle and hung over a wooden pole for 24 hr., after which both skin and

wool are in greatly superior condition to those treated conventionally. J. W. B.

Microscopic Investigation of Fur Bleaching and Dyeing (VIII p. 534).

### XIII—RUBBER; RESINS; PLASTICS

**Macromolecular Compounds. XLIV—Polyesters of certain Aromatic Dicarboxylic Acids.** V. V. Korshak and T. A. Soboleva. *Zvestiya Akad. Nauk S.S.R. Otdel. khim. nauk*, 526–530 (May–June 1952).

Ethylene glycol or diethylene glycol is condensed with a dicarboxylic acid of benzene (*o*-, *m*-, or *p*), diphenyl (*oo*-, or *pp'*), diphenylmethane (*pp*'), or *s*-diphenylethane (*pp'*), and the m.p. and solubility in various solvents, including dioxan, chlorform, alcohol, benzene, cresol, and cone.  $H_2SO_4$  of the 14 polymers obtained are recorded. The regularities observed are discussed. Some of the polymers, e.g. the linear polymers from ethylene glycol and *pp'*-diphenylcarboxylic acid or *s*-diphenylethane-*pp'*-dicarboxylic acid, are insol. in solvents other than hot cone.  $H_2SO_4$ , and it is pointed out that the commonly held view that only three-dimensional polymers are infusible and insoluble is incorrect. A. E. S.

**Solvents for Polycrylonitrile.** E. E. Walker. *J. Appl. Chem.*, 2, 470–481 (Aug. 1952).

The cohesive energy densities (C.E.D., g.cal./c.c.) of solvents for polycrylonitrile (mol. wt. 53,000) are reviewed. The best solvents, e.g. malononitrile, succinonitrile, cyclic ethylene carbonate, butyrolactone, tetramethylene sulphone, have high C.E.D. (~ 220), near that calculated for the polymer (~ 240), and solution is due to entropy factors; whereas *NN*-disubstituted amides (C.E.D. 116–170) are effective solvents owing to high affinity between solvent and polymer. Strongly hydrogen-bonded compounds, e.g. alcohols, amides, and phenols, are not solvents regardless of their C.E.D., but may contribute to the solvent power of other liquids, e.g. nitro-methane. Esters, ketones, and mononitrites are ineffective because of low C.E.D. E. C.

#### PATENTS

#### Rotproof Polyvinyl Chloride Compositions.

Monsanto.

BP 679,566 7

Polyvinyl resins containing predominantly combined vinyl chloride are rendered rotproof by incorporating with the resins (100 parts by wt.) a plasticiser (0–100), toluene-sulphonamide-formaldehyde condensate (1–25) or an *N*-alkylbenzene or toluene-sulphonamide (Alk of 1–8 C) (5–50), and Cu 8-quinoquinone (0.1–10.0). C. O. C.

#### Colourless Epoxide Hydroxymethylmelamine Condensates.

Monsanto.

USP 2,594,452

Condensing formaldehyde with melamine in presence of a boron compound or addition of a boron compound to a hydroxymethylmelamine results in stabilised hydroxymethylmelamines. These on condensation with epoxides yield colourless products. C. O. C.

#### Tung Oil Acrolein Condensation Products.

N.V. De Bataviafse Petroleum M.S.

BP 679,765

Quick-drying film forming materials are obtained by treating an addition product of an  $\alpha\beta$ -ethylenically unsaturated aldehyde (e.g. acrolein) and a salt, an amide, or an ester of a polyethylenic acid having two conjugate ethylene linkages (e.g. tung oil) with an ester-type condensation catalyst (e.g. aluminium propoxide). W. G. C.

Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Polymers or Copolymers of Conjugated Dienes (VIII p. 534).

Incorporating or Applying Dyes, Stabilisers, etc. in or on Reaction Products of Sulphur Dioxide with Macromolecular, Poly-unsaturated Compounds (VIII p. 534).

### XIV—ANALYSIS; TESTING; APPARATUS

#### Reaction of Sodium Nitrite and Sulphamic Acid—Indirect Gravimetric Determination of Nitrates.

R. C. Brasted. *Anal. Chem.*, 24, 1111–1114 (July 1952).

Soluble nitrates react rapidly with sulphamic acid— $NaNO_3 + NH_2SO_3H \rightarrow N_2 + NaHSO_4 + H_2O$

### XIV—ANALYSIS; TESTING; etc.

The reaction is made the basis of an indirect gravimetric determination of the nitrite, by measuring the loss in weight due to evolved nitrogen. Some nitrite is consumed in secondary reaction with hydrogen ions before it is able to react with the sulphamic acid, and this leads to the evolution of  $N_2O_3$  and  $NO_2$ . Since these oxides of nitrogen are found to be ca. 10% of the evolved gas, they lead to an appreciably high result, and a correction factor is applied. The method, which is given in full, may be used to determine the concn. of nitrite soln. between 0.1 and 0.5 M., and is applicable to soln. containing nitrates or coloured substances, or ions which interfere with iodometric analysis. Iron ( $Fe^{2+}$ ),  $SO_4^{2-}$ , and  $CO_3^{2-}$  interfere. J. W. D.

**Colorimetric Determination of Silver with *p*-Dimethylaminobenzylidenehydronine.** G. C. B. Cave and D. N. Hume. *Anal. Chem.*, 24, 1503–1505 (Sept. 1952).

**Sodium Aminoethoxide Titration of Weak Acids in Ethylenediamine—Application to Determination of Phenols in Coal Hydrogenation Oils.** M. Katz and R. A. Gleam. *Anal. Chem.*, 24, 1157–1163 (July 1952).

**New Azo Indicators of the Methyl Orange Series and the Relation between the Structure and the Transition pH of Azo Indicators.** V. I. Kuznetsov and G. N. Kosheleva. *J. Anal. Chem. U.S.S.R.*, 7, 56–62 (Jan.–Feb. 1952).

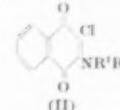
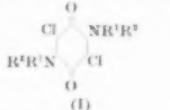
Indicators derived from 4-dimethylaminobenzene are of interest because their colour changes are not generally affected by complex-forming metals. The effect of substituents on the *pK* value is considered. 4-Substituted ( $CH_3$ ,  $OCH_3$ , Hal,  $SO_3H$ , COOH) have little effect, but substitution of halogen in the 2 position reduces the *pK* value considerably, owing, it is thought, to steric interference with the access of protons to the azo link. Four halogen derivatives of methyl orange (the 4'-sulphonic acid; pH range 3.1–4.5) are prepared and proposed for use as indicators at very low pH—2 chloro (pH 1.6–3.7); 2 bromo (pH 1.6–3.6); 2,5 dichloro (pH 1.4–3.2); 2,6 dibromo (pH up to 0.5). A. E. S.

**Determination of Reactive Hydrogen in Organic Compounds—A Review.** E. D. Ollerman. *Anal. Chem.*, 24, 1425–1439 (Sept. 1952).

Special procedures for sugars, cellulose derivatives, lignin, hydroxy acids, and primary and secondary amines are included in the discussion. There are 190 references to the literature. J. W. D.

**Characterisation of Amines by means of Halogenated Quinones.** N. P. Bui Hoi, R. Royer, and B. Eckert. *Rec. Trav. chim.*, 71, 1059–1064 (Sept.–Oct. 1952).

It is shown that the halogenoquinones, chloranil and (especially) 2,3-dichloro-1,4-naphthoquinone, may be used for the characterisation of primary and secondary amines, since the former reacts to give a 2,5-diamino-3,6-dichloro-*p*-benzoquinone (I) and the latter to give a 2-chloro-3-amino-1,4-naphthoquinone (II). 51 Deriv. are described.



H. H. H.

**Quantitative Spot Test on Filter Paper—Application to Alcohols and G and R Acids.** T. Kariyone, S. Shimizu, and Y. Hashimoto. *Nature*, 170, 422 (6 Sept. 1952).

A drop of soln. containing a few micrograms of substance under examination is placed in the centre of a filter paper (~ 11 cm. diameter), and a circular chromatogram is made by adding, dropwise, a suitable solvent to the drop, followed by a development period in a saturated atmosphere of the same solvent. Methanol and ethanol in admixture may be separately estimated by first forming their K xanthates, separating them as described by means of butanol saturated with 5%  $NaHCO_3$ , and examining the dried chromatogram under ultraviolet radiation, when the ethyl compound shows up as the outer ring. The zones may be marked by pencil, cut up, extracted with 5%  $NaHCO_3$ , and estimated electrophotometrically after

addition of Grote's reagent. By the same procedure, R acid and G acid may be separated and determined; in this case the diazonium salt of naphthionic acid is added to the chromatogram extracts, and the red dyes which develop are estimated spectrophotometrically. J. W. D.

**Analysis of Dyes for Man-made Fibres.** A. F. Schneid. *Amer. Dyestuff Rep.*, **41**, 565-572 (15 Sept. 1952).

Preliminary examination includes the separation and identification of diluent ('cutting agent'), purification of single dyes, detection of dye mixtures, and their separation by (i) chromatographic adsorption, (ii) methylation or acetylation, if one component is known, followed by regeneration of the original dye, and (iii) using solutions of different pH. Infrared or visible light spectograms show absorption bands characteristic of certain functional groups. Other useful physical tests include the determination of m.p. or sublimation range, and dyeing on multi-fibre cloth. Under the heading of chemical analysis, methods are discussed for the detection and estimation of constituent elements, with special emphasis on fluorine. An extensive section deals with the elucidation of dye structure. Amongst the tests described and discussed are—(i) Various reduction and re-oxidation tests; the behaviour of each dye class on reduction with Zn dust is shown in a table; (ii) identification of the azo grouping by  $\text{HNO}_3$  and  $\text{HCl}$ ; (iii) conc.  $\text{H}_2\text{SO}_4$  and the 'drowning' test to differentiate amongst azo dyes, and (iv) characterisation of diphenylamine dyes by the Schotten-Baumann reaction. Examples are discussed of the splitting and identification of the fission products of azo dyes. Azo dyes which are difficult to reduce may be split by fuming  $\text{HNO}_3$  into the diazonium nitrate of the amine base and the nitrated derivative of the coupling component originally used; the application and development of this general and important reaction are discussed. Other excellent reactions for identifying azo components, such as those of Rowles and Levin, and Palkin and Wales, have the disadvantage of not having been reviewed since the 1920s. Factors are discussed which are useful in the elucidation of anthraquinone dyes, e.g. only those which have  $\alpha$ -substituents have affinity for acetate rayon. Analytical procedures may be followed by a search of the literature. Absolute certainty can be reached only by ultimate synthesis of the dye, together with mixed m.p. and tests such as dyeing trials and the comparison of spectograms. J. W. D.

**Study of some Liposoluble Dyes by Chromatography with Alumina Discs.** M. Mottier and M. Pottier. *Mitt. Lebensm. Hyg.*, **43**, 118-123 (1952); *Chem. Abstr.*, **46**, 7942 (10 Sept. 1952).

A simple way of extracting and identifying colouring matter in fats is to drop the solution under examination from a pipette on to the centre of an alumina disc and elute with—pentane,  $\text{CHCl}_3$ , benzene,  $\text{CHCl}_3$ , ether, acetone, absolute alcohol, and water.

**Fixation of Chromatograms on Alumina Discs.** Idem, *ibid.*, 123-125.

As the dyes are very sensitive, they are best fixed by saturating them with paraffin wax. C. O. C.

**Colorimetric Test for the Elderberry Colouring Matter in Wine.** F. Wobisch and J. Schneyder. *Sitzungsber. Öster. Akad. Wiss.*, **161**, (3-4), 478-481 (1952).

The difference in behaviour towards the borate buffer solution of the anthocyanins of red wine (e.g. claret) and of the elderberry is established, and a test described for detection of elderberry sap in wine. H. H. H.

**Determination of Acidity in Offset Colours.** N. G. Goloshevich and L. S. Sukhanov. *Poligraf. Prozessirov.*, (11), 23-24 (1948); *Chem. Zentr.*, 1130 (1949); *Chem. Abstr.*, **46**, 7787 (25 Aug. 1952).

A method independent of the colour and degree of dispersion of the pigment consists in dissolving a 10 g. sample in 70 c.c. of benzene in a porcelain dish. The pigment is separated by adding some 80-90% alcohol and filtered off. The solvent is distilled off from the filtrate until the vol. is reduced to 30 c.c. The residue is then evaporated. A 0.3 g. portion of the residue is dissolved in neutral 1:4 alcohol-benzene and titrated with 0.1 N. alcoholic potash with Alkali Blue. The acidity of 1 g. of varnish is calculated by the formula  $K = aT/b$  in—amount of KOH in c.c.,  $b$  = wt. of sample;  $T$  = titre of alcoholic KOH in mg.). The acidity of the pigment is calculated from the

pigment: varnish ratio. This ratio is 1:0.3 for Cr pigments, 1:1.2 or 1:1.0 for lacquers, and 1:0.75 for Molar. C. O. C.

**Application of Phase-contrast Microscopy to Textile Problems.** M. Besse. *Bull. Inst. textile France*, (30), 79-89 (Jan. 1952).

Fundamentals of the method including a mathematical analysis are given. Applications to problems such as those involving the degradation of wool and the study of detergent solutions are discussed. G. L.

**Electron Microscope in Textile Research.** Y. Baillie. *Bull. Inst. textile France*, (30), 55-68 (Jan. 1952).

A description of the electron microscope and a summary of techniques used in the preparation of specimens for examination. The electron microscope has been applied in the study of textile fibres, colouring matters, and auxiliary products including soaps, resins, and emulsions. G. L.

**Measurement of Yarn Weight and Moisture Content by Transverse Vibration.** D. O. Sproule. *J. Textile Inst.*, **43**, T 455-T 462 (Sept. 1952).

Mass per unit length of a yarn is determined by noting the natural frequency of a measured length of yarn when subjected to known tension; as the moisture content alters, the tension is altered to provide the same natural frequency. The method is highly sensitive and is best applied to measure either changes in the mass per unit length which arise through changes in temp. or humidity, or changes in yarn uniformity when temp. and humidity are constant. J. W. B.

**Automatic Yarn Load Elongation Recorder.** F. B. Breazeale and H. D. Irvin. *Text. Research J.*, **22**, 549-552 (Aug. 1952).

An apparatus is described which subjects a length of yarn to a constant rate of elongation to break, or to repeated extension and retraction between either predetermined loads or elongations, with automatic recording. A. B.

**I.T.F.M. Abrasion Tester.** A. Marti. *Bull. Inst. textile France*, (32), 9-18 (April 1952).

The apparatus developed by the Institut Textile de France is described. The amount of fluff produced in the course of a test is weighed and plotted against time of treatment. The shape of the curves depends on the nature of the fibres as well as on the construction and finish of the fabric. The information obtainable from the graphs is claimed to be of value for the assessment of wearing qualities. G. L.

**Analysis of Staple Fibre Mixtures.** D. C. Fisher and E. D. Roberts. *Text. Research J.*, **22**, 309-312 (Aug. 1952).

Methods are described for the qualitative and quantitative analysis of staple fibre blends containing acetate rayon, dynel, wool, nylon, viscose rayon, cotton, Orlon, and Dacron in any combination. The qualitative analysis involves separation into groups according to density and the identification of members within a group by microscopic appearance. The quantitative analysis employs selective solvents. A. B.

**Modification of Norman Jenkins Method for Determination of Cellulose.** E. Bennett. *Anal. Chem.*, **24**, 1510-1511 (Sept. 1952).

The Norman-Jenkins method (*Biochem. J.*, **27**, 818-831 (1933)) is modified in that preliminary treatments are made with  $\text{NaClO}_3$  instead of alternately with alkaline  $\text{NaOCl}$  and  $\text{Na}_2\text{SO}_3$ ; all except the final separation may be made in the original container by means of a centrifuge and a microfilter stick. Compared with the original, the revised method gives slightly higher results, higher precision, and better removal of furfural and lignin. The use of acid  $\text{NaOCl}$  and  $\text{Na}_2\text{SO}_3$  in the final stages is recommended. J. W. D.

**Effects of Unfiltered Carbon Arc Light in Accelerated Weathering of Cotton and other Textiles.** J. D. Dean, C. M. Fleming, and R. T. O'Connor. *Text. Research J.*, **22**, 609-616 (Sept. 1952).

The degradative effects caused in cotton and other fibres by unfiltered carbon are light and by natural weather are compared. The results give no indication that the unfiltered carbon arc could be used in predicting the behaviour of chemically finished cotton in outdoor service,

although they suggested more possible value in the case of unfinished textile fibres.

A. B.

**Determination of Moisture in Fabrics by Measurement of their High-frequency Dielectric Constants—II.** T. Sakagami, T. Ebata, I. Kaino, Y. Tanaka, and E. Takagishi. *J. Soc. Textile Cellulose Ind.* (Japan), **7**, 369–372 (1951). **III.** T. Sakaguchi, T. Ebata, I. Kaino, Y. Tanaka, and E. Takagishi. *Ibid.*, **491**–494; *Chem. Abs.*, **46**, 6392 (10 July 1952).

**H**—A calibration curve with which to determine an unknown moisture content of the fabric was plotted from the dielectric constant of fabric determined when completely dry and when containing known amounts of moisture. The determinations were made by placing the fabric between two metallic-plate electrodes of a 500-kilocycle oscillating circuit with a secondary high frequency in the anode circuit to register any change with a galvanometer through a crystal rectifier.

**III**—By compressing the test fabric with 9.0 kg. or heavier weight the apparent density became almost constant and the dielectric constant was more stable, requiring less time (1–2 min.) for the determination. C. O. C.

**Standardisation of Methods of Testing Textile Auxiliary Agents—Rapid Determination of Residual Fat in Textiles.** H. G. Wagner. *Melland Textilver.*, **33**, 875–876 (Sept. 1952).

A simple method of obtaining a rough estimate of the fat content of a textile is to shake 5 g. of the material with 50 ml. of trichloroethylene for 3 min., allow to stand for 10 min., shake again for 3 min., and then allow a drop of the liquid to fall on a matt glass surface. The fat content is estimated by comparing the transparency of the spot formed when the solvent has evaporated with that of a spot produced by a soln. of known fat content. A. E. S.

**Estimation of the Crystalline Portion of Cellulose from Iodine Absorption Measurements.** K. Schwertassek. *Faserforsch. und Textiltech.*, **3**, 251–257 (July 1952).

In order to make the author's iodine absorption test for the degree of crystallinity of cellulose (see *J.S.D.C.*, **66**, 396 (July 1950); **67**, 126 (March 1951); **68**, 74 (Feb.), 235 (June 1952)) quantitative, estimates are made of the iodine absorption values for completely crystalline and completely amorphous cellulose. The former is considered to be close to the lowest value found for cotton hydrocelluloses, and an estimate of the latter is obtained from a consideration of the behaviour in the absorption test of highly amorphous analogues of cellulose, viz. acetate rayon and starch, and of cellulose itself (as rayon), using in each case a standard soln. and of iodine soln. and varying the amount of absorbent. Estimates made from absorption measurements of the crystallinities of cotton, mercerised cotton, wood cellulose, staple rayon, and high-tenacity rayon (Durafil) are 97%, 90%, 95%, 59%, and 75% respectively. A. E. S.

**Solubility Number of Linen as a Measure of its Degradation.** A. Caille and G. Cuvelier. *Bull. Inst. textile France*, **(30)**, 221–228 (Jan. 1952).

Full details of experimental work are given. It is concluded that the method described has great practical value, although the results obtained are not necessarily a measure of the degree of polymerisation. G. L.

**Rapid Analysis of Viscose Coagulating Liquor using Schwarzenbach Method—Volumetric Determination of Zinc and Sulphate Ion.** K. Ueno. *Anal. Chem.*, **24**, 1363–1364 (Aug. 1952).

The principal components of the liquor are—free  $H_2SO_4$ ,  $ZnSO_4$ , and  $Na_2SO_4$ . The free  $H_2SO_4$  is found by acidimetry.  $Zn$  is estimated by titration of the liquor with disodium ethylenediaminetetra-acetate (Versenate) using Eriochrome Black T as indicator (Schwarzenbach method). A similar titration is carried out after adding  $BaCl_2$  to the liquor, thus giving a measure of the total  $SO_4^{2-}$ . The  $Na_2SO_4$  is calc. from these three values. When the hardness of the water is appreciable it will affect the  $Zn$  estimation, and correction may be made by carrying out a blank titration after precipitating the  $Zn$  with ammoniacal  $Na_2S$ .

The method is advantageous in that a liquor may be fully analysed by only four titrations, three of which are made with the same Versenate soln. Five samples per

hour may be fully analysed. Equations are given by which each component may be calc. from the results of the four titrations.

J. W. B.

**Rapid Estimation of the Degree of Polymerisation of Cellulose by the Nitrate Method.** K. Eidelmann. *Faserforsch. und Textiltech.*, **3**, 301–302 (Aug. 1952).

The determination of the degree of polymerisation of cellulose from the viscosity of a soln. of the nitrate obtained by treating the cellulose with a mixture of  $HNO_3$ ,  $H_3PO_4$ , and  $P_2O_5$  is superior in accuracy to the cuprammonium method, in the course of which appreciable degradation occurs, but is time-consuming and tedious. A modification of the method, now described, can be readily carried out on large numbers of samples within the course of a few hours, and gives results agreeing with those obtained by the usual procedure.

A. E. S.

**Determination of Swelling Capacity of Viscose Rayon.** C. Rückstuhl and G. Durig. *Bull. Inst. textile France*, **(33)**, 9–20 (June 1952).

Factors affecting the gravimetric method for the estimation of swelling capacity are discussed. Important are—(i) the weight of fibres used, (ii) duration and speed of centrifuging, (iii) concentration of wetting agent and pH of the solution, and (iv) presence of spinning oils and the form and condition of the textile. Neither the liquor nor the duration and temperature of wetting out are significant.

G. L.

**Chemical Test for Modification of Wool.** M. van Overbeke, G. Mazingue, and P. Outreman. *Bull. Inst. textile France*, **(34)**, 53–70 (Aug. 1952).

Many testing methods depend on individual technique, and frequently are not reproducible by other workers. It is claimed that by means of the five tests described it is possible to identify the type as well as the extent of modification provided that a sample of the original wool is available for comparison. It is stated that if the degree of modification is small the dyeing of wool is more affected than are physical properties.

G. L.

**Rapid Estimation, especially Refractometric, of Residual Fat in Washed Wool and in Tops.** G. Nitschke. *Faserforsch. und Textiltech.*, **3**, 302–310 (Aug. 1952).

A review (12 references) is made of rapid methods of determining fat and oil in wool, particular attention being paid to the refractometric work of Clark and Thomas (see *J.S.D.C.*, **65**, 320 (June 1949); **66**, 504 (Sept. 1950)). Refractive-index determinations on solutions of wool fats of various origins show that errors due to variations in origin account for only part of the total experimental error in the refractometric method. The method is, however, sufficiently accurate for use in the control of scouring (which is contrary to the conclusions of Clark and Thomas (*loc. cit.*)). Wool-fat determinations are made on 18 samples of washed wool by extracting with ether (in a Soxhlet) or by Dickenson and Palmer's rapid method (see *J.S.D.C.*, **67**, 294 (July 1951)) and then estimating the extracted fat by direct weighing and by refractometry of solutions of the extract in ether or light petroleum. The Dickenson-Palmer method gives incorrect, low values for some types of wool, but refractometry of the Soxhlet extract gives generally satisfactory results. The recommended method is given with the necessary calibration curves.

A. E. S.

**Review of Shrinkage-testing Methods for Knitted Fabrics containing Wool.** E. G. Carter and A. N. Davidson. *J. Textile Inst.*, **43**, s 31–s 54 (Aug. 1952).

Felting properties of different types of knitted fabrics in stocks, wash wheel, domestic washers, rotary laundry machines, miniature stocks, and in hand-milling are described, and results are analysed statistically. Factors studied include pH values of relaxation liquors, wool load, machine speed, soap as compared with alkaline and acid perspiration test liquors, and presence of cracked soap.

J. W. B.

**Estimation of Cystine and Cysteine in Keratin.** J. B. Speakman and G. S. Park. *Bull. Inst. textile France*, **(30)**, 255–272 (Jan. 1952).

Difficulties arising in the Shimohara colorimetric method are indicated. The method is consequently modified. The first part of the paper gives optimum conditions for the estimation of cystine and cysteine in the hydrolysate

obtainable from a small specimen of wool. In the second part the experimental conditions necessary to avoid loss of cysteine are stated.

G. L.

**Adaptation of Classical Chromatographic Methods to the Separation of Amino Acids from Wool hydrolysed by Acid Chlorite.** J. Meybeck and C. Schurle. *Bull. Inst. textile France*, (30), 343-349 (Jan. 1952).

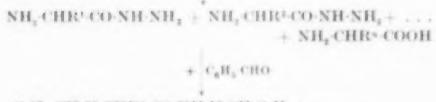
A detailed description of the techniques employed.

G. L.

**Hydrazinolysis of Proteins and Peptides—Method of Characterisation of Carboxy-terminal Amino Acids in Proteins.** S. Akabori, K. Ohno, and K. Narita. *Bull. Chem. Soc. Japan*, **25**, 214-218 (June 1952).

Treatment of peptides and proteins with anhydrous hydrazine causes breakdown of the peptide links, the carboxy terminal amino acids being liberated as the free amino acids, and the remainder as amino acid hydrazides. The latter are precipitated as insol. dibenzylidene derivatives by means of benzaldehyde, and the residual carboxy terminal amino acids separated and identified by partition chromatography. Results of experiments with glycyl-L-leucine, glutathione, insulin, and tyrocidin are given, and agree with those obtained by other methods.

$\text{NH}_2\text{CHR}-\text{CO}-\text{NH}-\text{CH}_2-\text{CO}-\text{NH}-\dots-\text{CHR}'-\text{COOH}$



A. J.

**Microdetermination of Thiol and Disulphide in Protein Hydrolysates by means of Phosphotungstate.** J. J. Kolb and G. Toennes. *Anal. Chem.*, **24**, 1164-1169 (July 1952).

The protein is hydrolysed in an evacuated sealed tube with a mixture of  $\text{H}_2\text{COOH}$  and  $\text{HCl}$  (under which conditions the cystine is sufficiently stable), and the SH is determined immediately in a portion of the hydrolysate, by the familiar phosphotungstate method; an acetate buffer is preferred to the citrate used in previous work, since the blue colour formed in its presence is more stable. A similar determination on another aliquot, which has been reduced with  $\text{Zn}$  under an atmosphere of  $\text{N}_2$ , gives, by difference, the SH resulting from reduction of the S-S-linkages. Extraneous reducers are estimated in the hydrolysate, using  $\text{HCHO}$  (preferred to  $\text{HgCl}_2$ ) as suppressor, and cysteine for the development of compensation colour. Each determination requires 100-300  $\mu\text{g}$ . cysteine or cysteine. Experimental details are given in full, and some weaknesses in previous variants of the procedure are discussed.

J. W. D.

**Benzidine Reagent in Paper Chromatography.** H. Miller and D. M. Kraemer. *Anal. Chem.*, **24**, 1371-1372 (Aug. 1952).

The potential interference of inorganic compounds, and some organic substances (particularly carbohydrates), in the identification of certain constituents of protein hydrolysates by paper chromatography using the benzidine reagent, is investigated. The colour reactions and  $R_f$  values of 22 inorganic and 19 organic compounds are listed; the behaviour of numerous other substances is referred to also, but in less detail.

J. W. D.

**Observations on the Birefringence and Refractive Index of Synthetic Fibres with special reference to their Identification.** A. N. J. Heyn. *Text. Research J.*, **22**, 513-522 (Aug. 1952).

Birefringence and refractive indices of synthetic fibres are detailed as measured by the polarisation colour and immersion methods. It is shown that these properties are much more consistent and characteristic for synthetic fibres than for natural fibres, and can therefore be used in the identification of the former. A simple polarising

device for making these measurements with an ordinary microscope is described.

A. B.

**Measurement of Adsorption of Anion-active Detergents by Materials commonly washed.** W. A. Fenster. *A.S.T.M. Papers on Evaluation of Soaps and Detergents, Spec. Tech. Pub.*, (115), 9-12 (1951); *Chem. Abs.*, **46**, 7347 (10 Aug. 1952).

Aqueous solutions containing surface-active ions can be titrated with an aqueous solution containing surface-active ions of opposite charge. Thus 12.5 g. desized cotton cloth was added to 50 ml. of 0.1% aq. synthetic detergent in a pint jar, which was then rotated in a Launderometer at 60°C. for 24 hr. 10 ml. of the detergent was then removed and put in a 250-ml. glass-stoppered Erlenmeyer flask, and 100 ml. distilled water added, followed by one drop of bromophenol blue soln. and 50 ml.  $\text{CHCl}_3$ . The soln. was then titrated with ethylpyridinium bromide soln., the flask shaken after each addition, the contents being allowed to settle, and the distribution of colour between the two layers noted. A convenient end point is equal colour density in the two layers. Low concentrations of some detergents leave wool darker than before, apparently because of redeposition. Detergent initially present in solution removes and suspends part of the soil but gradual depletion of the bath by detergent adsorption on the fibre, however, decreases the amount available for soil suspension, so that redeposition occurs.

C. O. C.

**Soiling Mixture for Detergency Evaluation.** R. E. Wag. *J. Textile Inst.*, **43**, T 515-T 516 (Sept. 1952).

An artificial soiling mixture for cotton fabrics is devised which results in improved correlation between laboratory tests using artificial soiling and laundry tests in which natural soiling is used.

J. W. B.

**Laboratory Performance Test for Detergents in Continuous Wool Scouring.** E. A. Leonard. *A.S.T.M. Papers on Evaluation of Soaps and Detergents, Spec. Tech. Pub.*, (115), 13-17 (1951); *Chem. Abs.*, **46**, 7348 (10 Aug. 1952).

5 g. Hanks of wool soiled with a special blend were passed through a line of beakers serving as bowls, being immersed for the same time as in scouring bowls on the plant and passed between squeeze rolls between each beaker, and the residual grease content was determined after drying. A rapid extraction method of grease determination provided data for plotting residual grease content against the cumulative weight of wool scoured. The customary detergent properties, e.g. surface tension, foam volume, wetting time, and Launderometer tests, do not correlate with actual works performance, but the above method can compare detergents on a quantitative economic basis, and compare also scouring conditions and control methods.

C. O. C.

**Evaluation of Soiled Test Pieces.** R. B. Mitchell. *A.S.T.M. Papers on Evaluation of Soaps and Detergents, Spec. Tech. Pub.*, (115), 3-8 (1951); *Chem. Abs.*, **46**, 7347 (10 Aug. 1952).

A study to determine the possibility of establishing "national standards" for soil removal as judged by soiled test pieces used in a commercial laundry. Three specific soiled test pieces were used; they were evaluated in a normal family whitework classification over a 20-wash test for soil removal. A single soiled test piece cannot adequately measure detergency as judged by soil removal under all working conditions. The adoption of "national" performance standards for soiled test pieces seems to be impracticable. The control-chart method of interpreting data is of value in determining control of specific washing formulae as related to individual plants. Soiled test pieces can be used advantageously by commercial laundries as long as their results are interpreted on the basis of the individual laundry only. A given soiled test piece cannot adequately measure the efficiency of the various detergents, washing formulae, or equipment as judged by soil-removal results obtained in actual works use.

C. O. C.

**Estimation of Fibre Degradation in Linen Bleaching.** W. Kind and H. Vollenbruck. *Textil-Praxis*, **7**, 716-720 (Sept. 1952).

The following properties of linen material are determined at each stage of the bleaching process—(1) yarn tensile strength, (2) degree of polymerisation, according to the authors' viscometric method (see J.S.D.C., **68**, 233 (June 1952)), (3) solubility number, (4) loss in wt. in the boil-off

used in the determination of (3) (2% NaOH, 4 hr.), (5) viscosity of the soln. formed by extracting the boiled-off material with 8% NaOH in the course of determining (3). Property (2) is to be preferred to (3) as an index of cellulose degradation; it is more sensitive to the changes occurring at the various stages of bleaching, and, moreover, the determination of (3) is shown to be subject to various uncertainties. (5) correlates more closely with (2) than with (3). Owing to the effect on (1) of the non-cellulosic components of the fibre, this property is not directly related to (2) or (3). A. E. S.

**Applied Spectrophotometry. I—Colour Matching with the aid of the "R" Cam.** R. E. Derby. *Amer. Dyestuff Rep.*, 41, P. 550, P. 558 (1 Sept. 1952).

The R cam on the G.E.C. recording spectrophotometer is used to plot  $(1 - R)^2/2R$ , the Kubelka-Munk function. The difficulties and advantages of this function as an aid to practical colour matching are described in detail. This is preceded by a theoretical discussion of the optical characteristics of turbid media and coloured textile materials.

A. H.

**Application of Colour Measurement in relation to Fibre Blending.** L. F. C. Friele. *J. Textile Inst.*, 43, p. 604, p. 611 (Aug. 1952).

The Kubelka-Munk formula cannot be extended so as to calculate the dyeing recipe from reflection curves of fibre blends, probably because the assumption of homogeneity of reflecting material is not valid. The empirically derived Noyel-Stearns formula gives a useful, though not wholly satisfactory, approximation to experimental data, and an attempt is accordingly made to develop a new formula on a theoretical basis. Experimental and theoretical reflectance curves are compiled for a series of felt made from different proportions of blue and white, and of red and white, fibres, and better agreement is found than when using the Noyel-Stearns formula. J. W. B.

**Colour Vision—A Retrospect.** W. S. Stiles. *Endeavour*, 11, 33-40 (Jan. 1952).

The history of theories of colour vision is summarised. Pictures in colour are given demonstrating colour blindness in the normal retina, Chevreul's discovery of the influence of coloured surrounds on a central colour, and Grassmann's laws of colour mixture. Other pictures show models of Munsell colour samples and the colours corresponding to different points in the C.I.E. colour chart. Rayleigh's anomaloscope is described, and the various forms of colour vision abnormalities are defined. L. P.

**Classification of Anomalies in Colour Vision.** G. N. Rautam. *Doklady Akad. Nauk S.S.R.*, 81, 815-818 (11 Dec. 1951).

A method of measuring the threshold of colour vision for red, green, and blue light, previously tested on 995 persons (*ibid.*, 74, No. 6 (1950)), has been applied to 305 persons. The thresholds ( $n_1$ ,  $n_2$ ,  $n_3$ ) are expressed as fractions of the most frequently occurring value for each colour in order to eliminate the scale of the instrument. In addition, the same persons were tested with a photoelectric form of Rayleigh's anomaloscope (*ibid.*, 73, No. 1 (1950)) to find the ratio of the intensities in a mixture of green and red light which they matched with yellow light from a standard interference colour filter ( $\lambda_{max}$  590 m $\mu$ ). Each experimentally found ratio is divided by the most frequent value (4.05) to give a coefficient  $A$ , which is  $\sim 0.95$  for persons weakly sensitive to red (protanomalous trichromats) and  $\sim 1.1$  for those weakly sensitive to green (deutanomalous).

A table of these coefficients is given for a sample of 10 representative of normal vision and all the 110 cases whose coefficients  $A$  deviated from the above norm (87 cases) and/or whose thresholds for any colour were not  $< 2.0$  (46 cases). Only 2 had  $n_1$  as high as 20 and 15 respectively (protanopes), 4 between 2.4 and 2.0, and only 1 as low as 0.6. For  $n_2$  there were 2 at 11.5 and 7.5 (deutanopes), while there were 18 between 2.8 and 2.0 and 5 as low as 0.3-0.4. On the other hand, the values for blue were not so large but there were more of them, 5 being between 4.3 and 3.1, 25 between 2.7 and 2.0, and 5 as low as 0.4-0.5. No correlation, however, was observed between threshold values and spectral sensitivity.

The following classification of colour vision is suggested—(1) Colour-weak—Trichromats with normal ratios of sensitivity to all three colours ( $0.95 < A < 1.1$ );

(2) Colour-blind—(a) Dichromats with normal sensitivity to two colours and complete or almost complete failure to perceive one colour (high threshold), via, (i) red-blind (protanopes), green-blind (deutanopes), and blue-blind (tritanopes); (b) monochromats, i.e. sensitive to only one colour. (2) Anomalous—Trichromats with an unusual distribution of sensitivity in the spectrum but with normal thresholds. (4) Colour-weak Anomalous—Trichromats with both abnormal distributions of sensitivity and high thresholds. L. P.

**Anomalous Colour Vision—Colour-filter Effect.** E. N. Yustova. *Doklady Akad. Nauk S.S.R.*, 81, 1051-1054 (21 Dec. 1951).

A Donaldson trichromatic colorimeter is used to measure the relative amounts of red, green, and blue light which different individuals mix in order to match monochromatic light over the whole range of wavelengths from 400 to 700 m $\mu$ . Using Maxwell's theory that colour defects are similar to the effects of interposing colour filters, the effective transmittances of such a filter at each wavelength are computed from the data of several protanopes and deutanopes. The numerical tests of the theory give very satisfactory results, and the effect of a yellow filter on normal vision is used as an experimental verification of the method. L. P.

**Retinal Colour Processes caused by Intermittent White Light.** K. Motokawa and M. Ebe. *Nature*, 170, 79-80 (12 July 1952).

Retinal response curves are reproduced to show the effects of flash duration of white light and interval between flashes on the colour perceived. J. W. B.

**Apparatus for Fastness Testing (Fading Lamps, Wash Wheels, Crockmeters).** SNV 9.5 0.58. Schweizerische Normen-Vereinigung. *Textil-Rundsch.*, 7, 368-369 (Aug. 1952).

The Rein fading lamp, containing low voltage discharge lamps fitted with suitable filters, is recommended for light fastness tests, since its emission spectrum closely corresponds to sunlight. G. L.

**Rapid Control Test to indicate Colour Fastness to Perspiration.** A.A.T.C.C. Tentative Test Method 71-52. *Amer. Dyestuff Rep.*, 41, P. 559 (1 Sept. 1952).

A simplified rapid test, not recommended to supersede TT 15-52 but useful for quick evaluation of dyed or printed samples. Spot specimen with test solution, cover with blotter, and heat to 250° F. under flat iron for 5 min. Criterion is transfer of colour to blotter. Acid test solution: 10 g. NaCl, 1 g. lactic acid (95%), 1 g. anhydrous disodium orthophosphate, per litre. Alkaline test solution: 10 g. NaCl, 4 g.  $(\text{NH}_4)_2\text{CO}_3$ , 1 g. anhydrous disodium orthophosphate, per litre. A. H.

**Rapid Control Test—Combined Colour Fastness and Shrinkage equivalent to Five Commercial Launderings.** A.A.T.C.C. Tentative Test Method 72-52. *Amer. Dyestuff Rep.*, 41, P. 560 (1 Sept. 1952).

A simple test, not to replace A.A.T.C.C. test No. 14-52, applicable to cotton and linen fabrics. Marked 5 in.  $\times$  5 in. specimens to which are attached 2 in.  $\times$  2 in. pieces of white fabric are laundered for 45 min. in a Launder-Ometer under conditions which increase the abrasion factor, i.e. larger metal containers, lower liquor ratio, and increased number of steel balls. Details of the washing and rinsing solutions, pressing, and evaluation of the test are given. A. H.

**Dye Contamination Detector.** Anon. *Dyer*, 108, 31-33 (11 July 1952).

The instrument is designed to measure the concn. in a colour box during printing of a soiling dye carried forward from a preceding box. A predetermined amount of radioactive phosphorus ( $^{32}\text{P}$ ) is included, as ammonium phosphate, in the colour paste likely to cause soiling. The box likely to be contaminated is fitted with two Geiger counters, one at each end, connected to a meter. The concn. of  $^{32}\text{P}$ , and hence of soiling dye, in the second box can then be read directly during printing. When this concn. reaches a critical level for soiling, the colour in the contaminated box is either changed, or fresh paste added.  $^{32}\text{P}$  emits only  $\beta$  radiations, which do not penetrate the normal containers for the liquids. The quantities required are sufficiently small for the radioactivity to be below the max. tolerance for human contact. A. J.

**Mercerisation of Yarn—Experimental Apparatus and Techniques.** C. F. Goldthwait, A. L. Murphy, I. W. Lohmann, and H. O. Smith. *Text. Research J.*, **22**, 549-548 (Aug. 1952).

Apparatus is described for the study of the mercerisation of cotton yarn in skeins and in lots weighing up to ~ 12 oz. A strain gauge is used for measuring the tension developed during mercerisation, and it is shown that this tension may reflect the initial properties of both yarn and cotton as well as process conditions. A. B.

**Detection of Melamine Resins in Cellulosic Fibres and Fabrics.** H. A. Wannow. *Melliand Textilber.*, **33**, 758-761 (Aug. 1952).

Known methods of locating melamine resins in yarns and fabrics, using acid dyes or Tollen's reagent as stain, are discussed, and their application is illustrated by photomicrographs. These methods are of little value for detecting variations in the distribution of resin over a fabric. This is best done by dyeing the fabric with an acid dye, e.g. Suprano Scarlet FG:N, and observing the variation in the depth of dyeing. Photographs are shown of the results of a test on a fabric in which the resin is predominantly on one face and of a test to determine the variation of resin fixation with curing temp. A. E. S.

**Test Method for Resistance of Wool and other Keratinous Materials to Moths and Beetles.** SNV 95.9.10. Schweizerische Normen-Vereinigung. *Textil Rund.*, **7**, 329-330 (July 1952).

**Estimation of the Distribution of the Substituents in partly Substituted Carboxymethyl Celluloses.** T. E. Timell. *Scand. Papperstidn.*, **55**, 649-660 (15 Sept. 1952).

A method has been developed for estimating the distribution of the substituents in anhydroglucosidic units of partly substituted carboxymethyl cellulose, a commercial grade being degraded with  $H_2SO_4$  and the sugar mixture formed isolated after selective neutralisation with either an anion exchange resin or  $Ba(OH)_2$ . A new method for determining the number of primary OH groups in the 6-position in cellulose ethers, involving oxidation with  $HIO_4$  of the corresponding glucose mixture and estimation with dimedone of the  $HCHO$  formed by oxidation of the glycid group in the 5-6 position, gave reliable and reproducible values. Tsylyatkin iodination and tritylation gave respectively considerably lower and slightly higher values than the  $HIO_4$   $HCHO$  method. The number of mono-, di- and tri-substituted anhydroglucosidic units was determined by selective extraction with various solvents of the corresponding methyl esters of the carboxymethylated glucose mixture. The complete absence of anhydroglucosidic units substituted in both the two secondary positions was indicated. S. V. S.

**Analysis and Chemical Composition of Naphthalene-sulphonic Acid Syntans.** D. R. May, F. D. Thayer, and J. D. McGovern. *J. Amer. Leather Chem. Assoc.*, **47**, 482-491 (July 1952).

Methods for determining polarographically the sulphate content of naphthalene-sulphonic acid syntans are outlined, together with a rapid technique for determination of ammonium nitrogen. A. S. F.

**Metal Indicators. VI. A Nitrophenol forming Complexes with Metal Cations.** (IV p. 521).

**Trityl Derivatives of Amines. I. N-Triphenylmethyl Derivatives of Aromatic Primary Amines.** (IV p. 521).

**Infrared Absorption Spectra of Porphyrins.** (IV p. 524).

**Method for Studying the Effect of Humidity on the Cross-sectional Swelling of some Common Fibres.** (VI p. 529).

**Viscometric Study of the Molecules of Sodium Dodecyl Sulphate in Dilute Solutions.** (VII p. 531).

**Controlled Vat Dyeing. II. Instrumental and Analytical Measurement.** (VIII p. 532).

**Problems presented by the New Fibres [Identification and Dyeing].** (VIII p. 533).

**Urea Formaldehyde Fixation on Viscose Rayon.** (X p. 535).

**Acid Strength of Glucuronic Acid in Comparison with that of Oxycelluloses.** (XI p. 537).

**Reactive Groups of Collagen. I. Reactions of the  $\alpha$ -Amino Group of Lysine.** (XII p. 538).

## XV—MISCELLANEOUS

**Studies in Chemical Documentation.** G. M. Dyson. *Chem. and Ind.*, **67**, 676-684 (12 July 1952).

Organic compounds are symbolised for indexing and reference purposes, each symbol consisting of a "miform index" followed by a "Dyson index"; each comprises three sets of numbers, separated by full stops, and the two indexes are separated by a colon. To obtain the miform index the empirical formula of the substance is cited with the elements in a set order, grouped as—

Other elements P I F Cl Br S N O C H  
(3) (2) (1)

Elements in (1) and (2) are shown simply as the number of atoms present in the molecule (when this is 10 or more, the second figure is given as a subscript). A zero on the extreme left of the symbol means that no element further to the left in the above sequence is present, whereas a zero anywhere else means that the element in that position is absent. In (3), the elements are shown by their conventional symbols with subscript numbers (as in the empirical formula itself). The Dyson index further sifts the compounds according to their structural features. Of the three sections comprising it, (1) and (2)—numbered from the colon—show the number of certain functional attributes present, in a fixed order, whilst in (3) the numbers given are the ordinal numbers of the attributes in the order in which they are listed by the author. Only the first three positive attributes are given in each section, the numbering beginning with the first of these and, except in section (3), with intervening attributes which are absent shown as zeros. The three sections of the Dyson index consist of the following functional attributes, in the order cited—

(1) Number of rings larger than 6	(2) Number of heterocyclic atoms
Number of rings smaller than 5	Number of triple bonds
Number of rings of 6	Number of double bonds
Number of rings of 5	Number of ethyl groups
Number of aromatic rings	Number of methyl groups
(3) COOH or ester group	
CO, keto only	
CHO	
NH <sub>2</sub> or amide	
NO or NO <sub>2</sub>	
O, Alk	
More than one ring substituent	
Discontinuous carbon skeleton	

Together with the cipher system already published by the same author, and provisionally accepted as an international system, these symbols provide a rapid and convenient method for tracing compounds without recourse to trivial names. The above arrangement of elements brings together compounds of similar structure instead of dispersing them, as is the case when an index is based on increasing numbers of carbon and hydrogen atoms in the first place. Amongst the 120 examples listed by the author, together with their Dyson ciphers and indexes, the following appear—

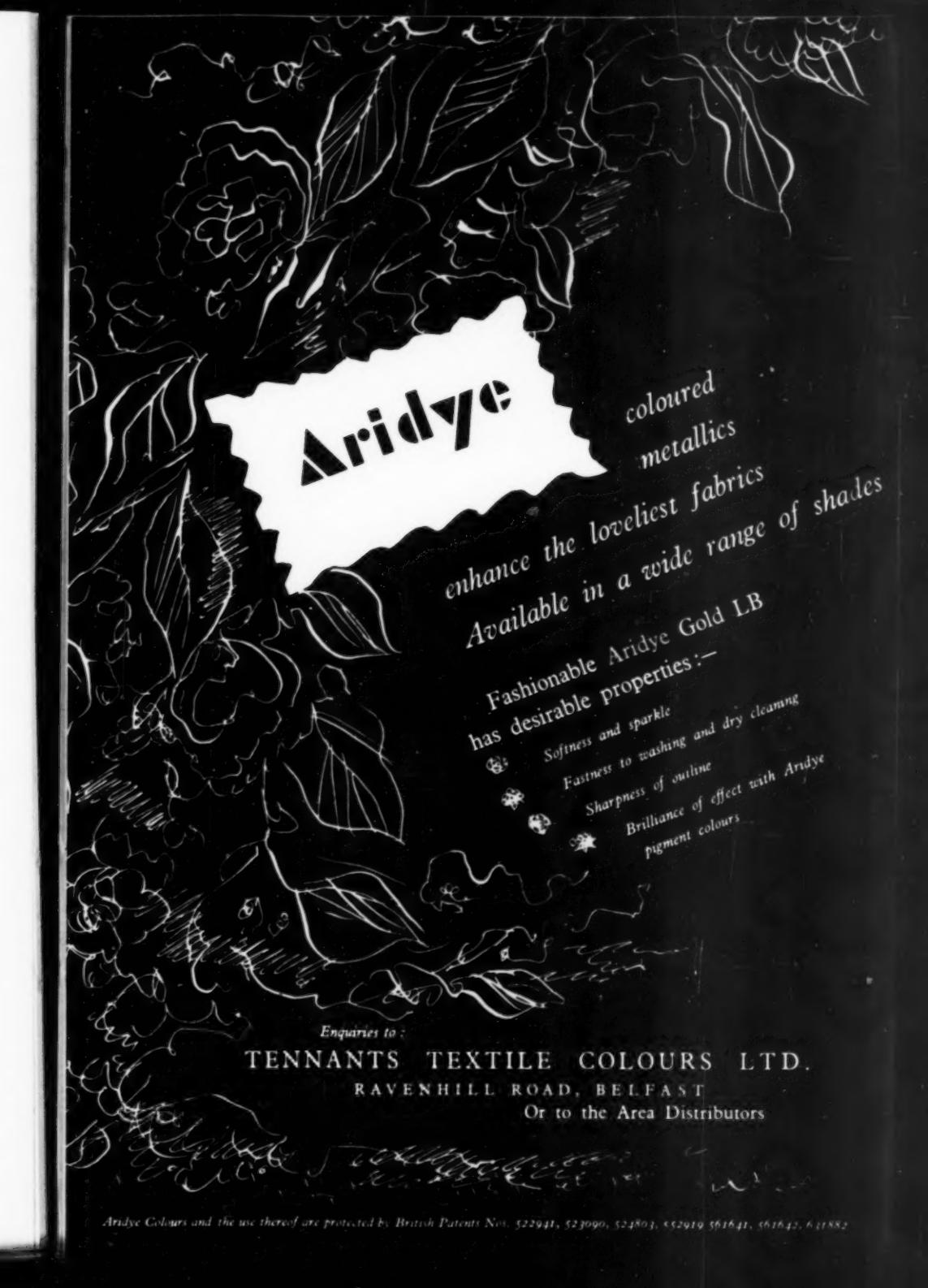
Stilbene	$C_6H_5CH=CH-C_6H_5$	0.1 <sub>2</sub> 1 <sub>2</sub> 2.21.100.0
Fluorene	$CH_2-C_6H_5-C_6H_5$	0.1 <sub>2</sub> 1 <sub>2</sub> 1.23.0.0
Styrene	$C_6H_5CH=CH_2$	0.88-11.100.0

J. W. D.

**Production of Compendia.** J. W. Duarte and A. E. Stubbs. *J.S.D.C.*, **68**, 512 (Dec. 1952).

**Indexing by Empirical Formula.** J. H. Fletcher and D. S. Dubbs. *Chem. and Ind.*, **931**-932 (20 Sept. 1952).

Agreement is expressed with Dyson (above) that C and H content of organic compounds is of subsidiary importance in indexing. Unlike Dyson, the authors place the empirical formula in alphabetical order, and do not translate them into numerical ciphers; they hope to publish details of their system soon. J. W. D.



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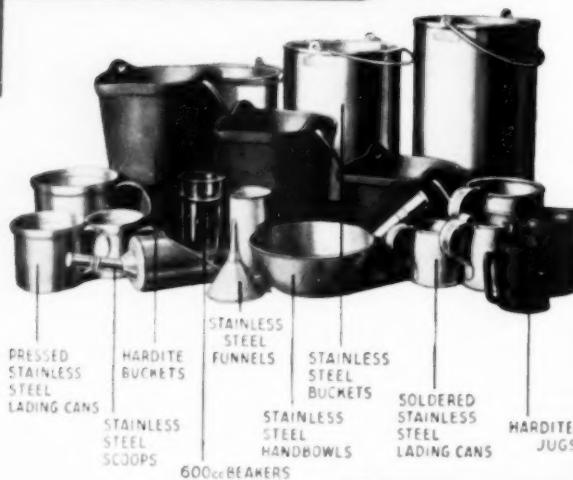
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FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page xii*

## LEEDS JUNIOR BRANCH

*All meetings to be held in the Colour Chemistry Lecture Theatre, The University, Leeds, 2 on Tuesdays at 4.0 p.m.*  
1953

- 20th Jan. J. Boulton, Esq., M.Sc.(Tech), F.R.I.C., F.T.I. (Courtaulds Ltd.). Title to be announced
- 17th Feb. G. G. Taylor, Esq., B.Sc., A.Inst.P. and J. C. Brown, Esq. (The Clayton Aniline Co. Ltd.). *The Use of Microscopy in Textile Dyeing and Finishing*
- 3rd March D. Hanson, Esq., B.Sc., F.R.I.C. (J. Crowther & Sons Ltd.). *A Chemist in the Woollen Industry*

## WEST RIDING SECTION

*All meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated*

- 1953
- Friday 16th Jan. LADIES' EVENING
- Thursday 29th Jan. Lecture. Details later
- Thursday 12th Feb. D. P. Raper Esq. (Imperial Chemical Industries Ltd.). *Wool Dyeing Faults and their Correction*
- Monday 23rd Feb. N. R. Hjort, Esq. *Water Treatment*. (Joint lecture with the Halifax Textile Society). Alexandra Cafe, Halifax

West Riding Section — *continued*

- 1953
- Thursday 26th Feb. J. V. Summersgill Esq. (Geigy Co. Ltd.). Title later
- Tuesday 3rd March Dr. H. Baines (Kodak Ltd.). *Colour Photography*. The University, Leeds
- Thursday 12th March Dr. J. F. Gaunt (Patons & Baldwins Ltd.). *A Study of the Afterchrome Process of Dyeing Wool*
- Thursday 26th March ANNUAL GENERAL MEETING

## BRADFORD JUNIOR BRANCH

*All meetings held in the Bradford Technical College at 7.15 p.m.*

- 1953
- Monday 19th Jan. J. W. Fisher, Esq., B.Sc., F.R.I.C. (British Celanese Ltd.). *Synthetic Fibres*
- Wednesday 4th Feb. Visit to Messrs. Montague Burton Ltd., Hudson Road Mills, Leeds
- Wednesday 18th Feb. T. Green, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of pH and its Importance in Relation to Textile Processing*
- Thursday 26th Feb. JUNIOR BRANCH DANCE at the Queens Hall
- Friday 13th March STUDENTS' ANNUAL COMPETITION EVENING
- Thursday 26th March FILM EVENING

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**Volume II, October 1952, xvi, 737 pages, illustrated**

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Acid Anthraquinone Dyes  
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Sulfur Dyes  
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Cyanine Dyes  
Miscellaneous Dyes  
The Action of Light on Dyes and Dyed Fibers  
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Nitro Dyes  
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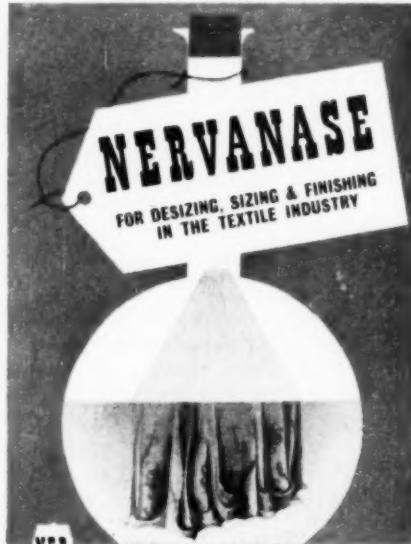
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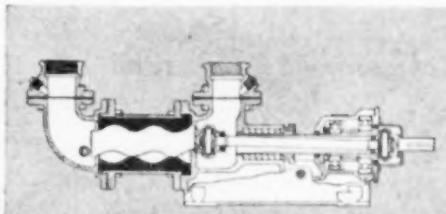
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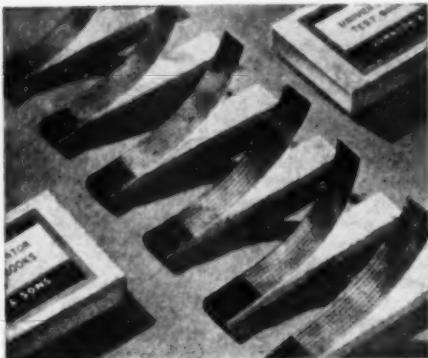
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